

1-1-1996

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Kingroungpet, Benjaporn; Phadungcharoen, Thatree; Ruangrunsi, Nijsiri; and Sekine, Toshikazu (1996) "Constituents of *Artocarpus gomezianus* Leaves," *The Thai Journal of Pharmaceutical Sciences*: Vol. 20: Iss. 1, Article 7.

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Constituents of *Artocarpus gomezianus* Leaves

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Abstract : Examination of the ethanol extract from the leaves of *Artocarpus gomezianus* (Moraceae) revealed the presence of a long chain hydrocarbon, 1-dotriacontanol, a steroid; β -sitosterol, two triterpenoids; lupeol-3-acetate and simiarenol, and phenolic glycoside; arbutin. The identification and structure elucidation of these compounds were established on the basis of detailed spectroscopic analyses.

Key words : *Artocarpus gomezianus*, Moraceae, 1-dotriacontanol, β -sitosterol, lupeol-3-acetate, simiarenol, arbutin

Introduction

The genus *Artocarpus* belongs to the tribe Artocarpeae of the family Moraceae. This genus consists of *ca* 50 species distributed in Ceylon, India and South China to the Solomon Islands, absent from Australia and New Caledonia (1). *Artocarpus gomezianus* Wall. ex Trec. is a medicinal tree locally known in Thai as "Haat nun" (2). The bark of this plant is claimed function the treatment of burns (3). These have been previous reported of phytochemical studies on only heartwood of *A. gomezianus* (4), and in this article we report the structural elucidation of five constituents isolated from the leaves of this plant.

Result and Discussion

The residue from ethanol extraction of the leaves was concentrated and separated to reveal five components by quick column chromatography as described in Experimental section. The first and least polar compound **1**, was obtained as white crystals and identical as 1-dotriacontanol. The IR (3424 cm^{-1}) and NMR data suggested that **1** was a long chain alcohol. The EIMS exhibited a peak at m/z 449 corresponding to $[\text{M-OH}]^+$ and showed characteristic fragmentation pattern of long chain hydrocarbon compound by the clusters of peaks, and the corresponding peaks of each cluster were $14(\text{CH}_2)$ mass units apart because the loss of $\text{CH}_2=\text{CH}_2$ (5). The proposed carbon signal assignments of carbons of **1** are predicted by calculation using the additivity rule.

The second component was obtained as colorless needles. The EIMS of **2** exhibited a molecular ion peak at m/z 414 suggesting a molecular formula of $\text{C}_{29}\text{H}_{50}\text{O}$. It was identified as β -sitosterol, by analysis its ^{13}C NMR spectrum which revealed the signals of 3 quaternary, 9 methine, 11 methylene and 6 methyl carbons. The signal assignments of carbons of **2** are supported by comparison with the data of β -sitosterol previously reported by Ogura, Cordell and Farnworth (6).

The third compound was obtained as colorless needles. The EIMS of **3** showed a molecular ion peak at m/z 468 suggesting a molecular formula of $\text{C}_{32}\text{H}_{52}\text{O}_2$. This could be assigned as the known pentacyclic triterpene lupeol acetate, by analyses of its ^{13}C NMR spectrum which suggested the presence of 8 methyl, 11 methylene, 6 methine and 7 quaternary carbons including 1 ester carbonyl carbon. The carbon assignments of **3** are confirmed by comparison of revealed data of lupeol acetate previously reported by Shieh and Lin (7) and Sholichin *et al* (8).

The fourth compound was obtained as colorless needles. The EIMS spectrum of **4** exhibited a weak molecular ion peak at m/z 426 and the accurate mass was consistent with the molecular formula $\text{C}_{30}\text{H}_{50}\text{O}$. It could be assigned as a known pentacyclic triterpene, simiarenol that was 3β -hydroxy-E:B-*friedo*-hop-5-ene (9) by the analyses of its ^1H and ^{13}C NMR spectra. ^{13}C NMR spectrum indicated thirty carbon signals which were the signals of 8 methyl, 9 methylene, 7 methine and 6 quaternary carbons. The signal

assignments of protons and carbons are supported by comparison with the data previously reported by Chakravarty (10) and Tulloch (11).

The fifth compound was obtained as colorless needles. The HR-FAB-MS (dithiodiethanol + NaCl) of **5** displayed a pseudomolecular ion $[M + Na]^+$ at m/z 295.0787, corresponding to the formula $C_{12}H_{16}O_7 Na$ (Calcd. for 295.0793 a.m.u.), and therefore suggesting a molecular formula of $C_{12}H_{16}O_7$. The UV absorption at λ_{max} 224 (log ϵ 3.82) and 286 (log ϵ 3.32) showed the characteristic of phenolate with *p*-substituent of hydroxy group. This was determined as 4-hydroxyphenyl- β -D-glucopyranoside, arbutin, by analyses of its 1H and ^{13}C NMR spectra. The 1H NMR spectrum provided the signals of 4H aromatic protons (A_2B_2 type) and signals of one set of β -glucopyranose unit. The ^{13}C NMR spectrum suggested the presence of 6 aromatic carbons and 6 oxygenated sp^3 carbons. The DEPT spectrum showed that **5** has 2 quaternary carbons, 9 methine carbons and 1 methylene carbon. The 1H NMR and ^{13}C NMR spectra (in pyridine- d_5) of **5** are identical with the data previously reported by Kubo and Ying (12).

Materials and Methods

Plant material : The leaves of *Artocarpus gomezianus* Wall. ex Trec. (Moraceae) were obtained from the Faculty of Pharmaceutical Sciences, Chulalongkorn University, Bangkok, Thailand, in November, 1993. The plant material was authenticated by comparison with the herbarium specimen in the Botany Section, Technical Division, Department of Agriculture, Ministry of Agriculture and Cooperative. A voucher specimen was deposited in the herbarium of the Faculty of Pharmaceutical Sciences, Chulalongkorn University, Bangkok, Thailand.

Instrumental : UV spectra were obtained on a Milton Roy Spectronic 3000 Array spectrometer, IR spectra on a Shimatsu IR-440 infrared spectrometer, mass spectra on a Fison VG Trio 2000 quadrupole mass spectrometer operating at 70 eV and the HR-FAB-MS was measured with a JEOL HX-110 mass spectrometer. 1H NMR (500 MHz), COSY, ^{13}C NMR (125 MHz) and DEPT spectra were recorded in $CDCl_3$, $DMSO-d_6$ and pyridine- d_5 , with TMS as internal standard, employing a JEOL JMN-A 500 spectrometer.

Isolation of 1-5 : The dried powdered leaves of *Artocarpus gomezianus* (1.8 kg) was macerated twice for three days with 95% ethanol and filtered. After combination, the extracts were evaporated *in vacuo* to yield 146 g of syrupy mass. The crude ethanol extract was purified by quick

column chromatographic technique using a sintered glass filter column of silica gel (20 x 4 cm). A gradient of chloroform in hexane, chloroform and a gradient of methanol in chloroform were used as eluents respectively. Fractions of 300 ml were collected and examined by TLC. Fractions 62-78 afforded 156 mg (0.00867%) of **1** and 79-98 afforded 72mg (0.004%) of **2** after recrystallization from chloroform and hexane respectively. Fractions 49-54 after drying, was separated using column chromatographic technique with a gradient system of 1-50% chloroform in hexane as an eluent to afford 69 mg (0.00383%) of **3**. Fractions 55-61 was fractionated by column chromatographic technique with a gradient of chloroform in hexane as eluent and further eluted with hexane : ethylacetate (49:1) to afford **4**, 25 mg (0.00139%). Fractions 142-145 was isolated by quick column chromatographic technique using chloroform, ethylacetate and 5% to 10% methanol in ethylacetate as eluents to afford **5**, 3.02 g (0.168%).

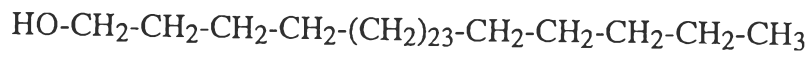
1-Dotriacontanol **1** obtained as white crystals; EIMS m/z (rel.int.) 449(0.37), 421(0.26), 393(7), 365(7), 364(3), 209(2), 195(3), 181(3), 167(4), 153(6), 139(9), 125(18), 111(36), 97(66), 83(72), 69(67), 57(100), 55(64), 43(100); IR ν_{max} (KBr) cm^{-1} 3424, 3298, 2918, 2849, 1473, 1463, 1062, 720 ; 1H NMR (500 MHz, $CDCl_3$) 0.88 (t, $J=7.2$ Hz), 1.25 (br.s), 1.55 (m), 3.63 (t, $J=7.2$ Hz) ; ^{13}C NMR (125 MHz, $CDCl_3$) 14.06(C-10), 22.65(C-9), 25.71(C-3), 29.31 (C-7), 29.39(C-4), 29.55(C-6), 29.65(C-5), 31.87(C-8), 32.78(C-2), 63.07(C-1)

β -Sitosterol **2** obtained as colorless needles ; EIMS and ^{13}C NMR, see ref.6

Lupeol-3-acetate **3** obtained as colorless needles ; EIMS and ^{13}C NMR, see ref. 7,8

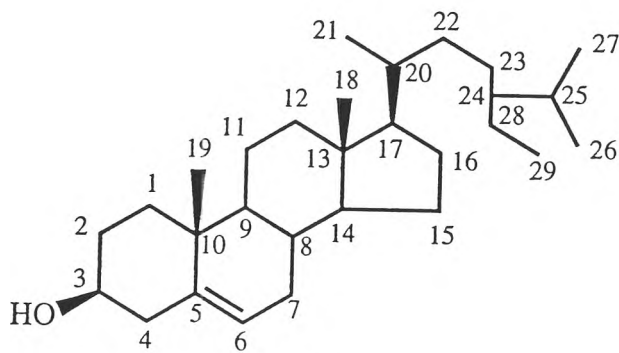
3 β -Hydroxy-E : B-friedo-hop-5-ene **4** obtained as colorless needles; EIMS m/z (rel.int.) 426(M^+ ,4), 411(2), 408(3), 274(100), 259(90), 245(13), 231(26), 205(13), 175(75), 152(42), 134(66), 122(56), 107 (40), 95(50), 81(40), 69(39), 55(37) ; IR ν_{max} (KBr) cm^{-1} 3508, 2931, 2868, 1650, 1470, 1545, 1384, 1052, 831, 818; 1H NMR and ^{13}C NMR, see ref. 10, 11

4-Hydroxyphenyl- β -D-glucopyranoside **5** obtained as colorless amorphous; HR-FAB-MS (dithiodiethanol + NaCl) m/z (rel.int.) 295(29), 273(16); EIMS m/z (rel.int.) 110 (100);UV λ_{max} (MeOH) nm (log ϵ) 224(3.82), 286(3.32); IR ν_{max} (KBr) cm^{-1} 3372, 3292, 3204, 1611, 1513, 1223, 1106, 1080, 1050, 1027, 1016, 833; 1H and ^{13}C NMR, see ref. 12

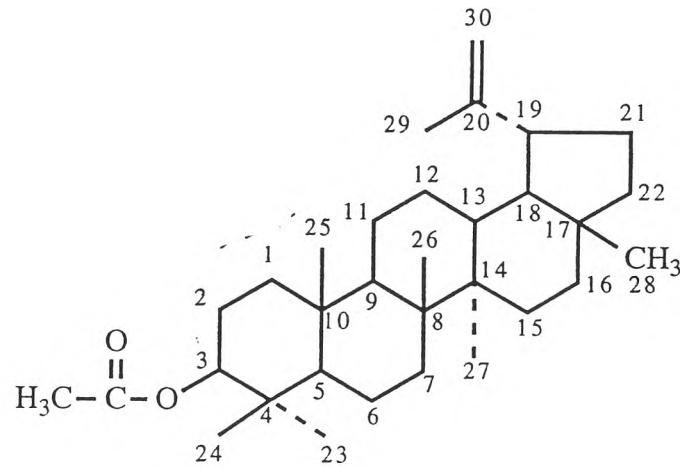


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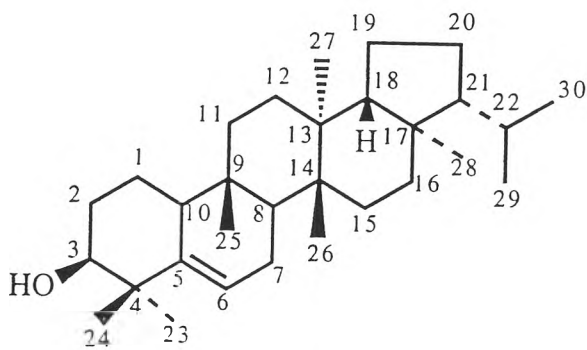
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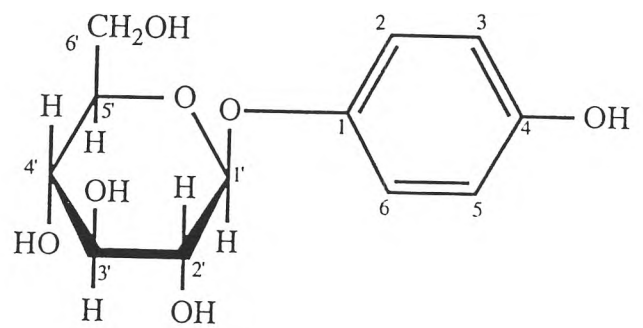
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3



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5

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