Prenylated Flavone of the Bark of *Artocarpus elasticus* from Alor Island-NTT Indonesia

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ABSTRACT

A new prenylated flavone, *artoindonesianin A-3*, were isolated from the bark of *Artocarpus elasticus*, an endemic plant from Alor Island – Nusa Tenggara Timur. This study revealed that *A. elasticus* is the potential source of flavonoids. The structure of compound was determined by UV, IR and NMR spectrometers.

Keywords: *Artocarpus elasticus*, flavon, *artoindonesianin A-3*

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1. Introduction

Artocarpus is an important genus of Moraceae family. Some species of this plant have been reported to have secondary metabolites, especially 3-prenylated flavonoid derivatives. The compounds with the basic framework of prenylflavonoid, which is a prenylated flavones at C-3 and the oxygenated at 2', 4' or trioxxygenated at 2', 4' and 5' to ring B (Ersam 2002). Some previous researches have been reported several prenylated flavone derivatives from *Artocarpus elasticus*, such as *artelasticin* (1), *artocarpesin* (2), (Kijjoa et al., 1996), *artobiloxanthone* (3) (Jayasinghe, 2008), *artonindonesianin V* (4) (Syah, 2004), *artoindonesianin A-2* (5), *Artoindonesianin A-3* (6) (Syah, 2006).

Artocarpus elasticus is one of species from this genus, which known as Tong-tong in Alor Island – Nusa Tenggara Timur. This plant has various applications, such as the trunk is used as building material, the leather sap is used as glue and the leaves are used as mosquito repellent. However, the application of *Artocarpus elasticus* as a medical plant has not known.

2. Materials and Procedures

2.1. Materials

The bark of *Artocarpus elasticus* was obtained from Alor Island – Nusa Tenggara Timur. A specimen was identified in LIPI Purwodadi (10 Feb 2015, the protocol refers to Backer at al., 1965). The chemicals used in the experiments included CH₂Cl₂, ethyl acetate, methanol, distilled water, acetonitrile, etc. The equipments included TLC, Spectrophotometer UV-Vis, NMR, and FT-IR.

2.2. Experimental Procedures

2.2.1. General procedure

Melting point was determined using Fisher John-melting point Apparatus. UV and IR spectra were measured with UV Pharma spec-1700 Shimadzu and FT-IR PRESTIGE 21 (SHIMADZU) spectro- photometers. The ¹HNMR 500 MHz and ¹³CNMR 125 MHz spectra were recorded with JEOL-Nuclear Magnetic Resonance spectrometer. Column chromatography was carried out using Merck Silica gel 60 GF254 and TLC analysis on precoated Silica gel plates (Merck Kiesel gel 60 F254, 0.25 mm). Silica gel Merck Si gel 60 GF254 is used for column.

2.2.2. Plant material extraction and isolation

Dry powder of *A. elasticus* bark (5 Kg) macerated in ethylacetate. The ethyl acetate extract (30 g) was fractionated by column chromatography using CH₂Cl₂, EtOAc and MeOH based on increasing polarity to give 6 fractions (A-F). There was 3 fractions (B, C and D) which have relatively the same R_f value, so that the combined and further fractionated by column chromatography method using MeOH:methylene chloride based on increasing polarity. Results from the fractionation combined to give 6 fractions (A1-A6) are monitored by TLC. There are 2 fractions (A2-A3) which have relatively the same R_f value, so that the combined and further fractionated by column chromatography method using eluent of methanol in methylene chloride: n hexane to give 4 fractions (B1-B4). And then the two fractions (B2-B3) were refractionated to give 5 fractions (C1-C5). The resulting precipitated fraction of C4 was filtered and then purified by re-crystallization to give compound 1 (36 mg).

Fig. 1. Molecular structure of the compounds isolated from the bark of *Artocarpus elasticus*Table 1. Data of ^1H -NMR and ^{13}C -NMR (DMSO) of Compound 1 and *Artoindonesianin A-3*

Position	Compound 1		Artoindonesianin (Syah, 2006)	
	δH (ppm)	δC (ppm)	δH (ppm)	δC (ppm)
2		163.6		161.7
3		111.4		111.4
4		179.6		181.0
4a		103.4		105.9
5		161.2		157.0
6		110.5		105.4
7		160.4		159.5
8		94.2		95.6
8a		156.6		156.7
9	2.36 (dd, 15; 5); 3.82 (dd, 5)	21.6	2.42 (dd, 15.9; 6.5); 3.40 (dd, 15.9; 1.7)	22.2
10	3.83 (brd, 5)	37.0	4.00 (brd, 6.5)	37.9
11		144.0		145.2
12	4.14 (m); 4.60 (m)	110.6	4.29 (m); 4.65 (m)	111.8
13	1.72 (m)	21.2	1.77 (m)	21.9
14	6.71 (dd, 10)	116.7	6.65 (dd, 10.0)	115.9
15	5.73 (d, 10)	129.2	5.73 (d, 10.0)	129.1
16		76.9		78.4
17	1.43 (s)	27.5	1.44 (s)	28.3
18	1.41 (s)	27.5	1.44 (s)	28.3
1'		108.0		106.4
2'		144.7		151.3
3'	6.69 (s)	98.6	6.50 (s)	103.7
4'		156.6		151.0
5'		136.0		136.7
6'		129.2		129.3
5-OH	13.12	-	13.67	-

3. Results and Discussion

Compound 1 was also isolated as a yellow powder with melting point of 143-145°C. The UV spectrum shows

absorption maxima at 252 and 356 nm. Addition of NaOH causes the tape I underwent a bathochromic shift from 356 nm to 368 nm which indicated the presence of free hydroxyl

groups. In addition, the UV spectrum of compound 1 showed bathochromic shifts from 356 nm to 425 nm upon addition of AlCl_3 .

The IR absorption bands were observed at 3529 and 3425 cm^{-1} which the characteristic of O-H absorption. Absorption at 2974, 2926, 2856 cm^{-1} typical for C-H aliphatic, absorption at 1654 cm^{-1} showed a carbonyl group while the absorption 1614, 1568 and 1450 cm^{-1} show the aromatic C-H. The ^1H NMR spectral parameters of 1 (Table 1) showed a similarity to the *artoindonesianin A-3* and revealed the presence of a singlet for a chelated hydroxyl group (δH 13.12), a series of signals (1.72; 2.36; 3.82; 3.83 4.14 and 4.60) assigned to a spin system $\text{CH}_2\text{--CH--C}(\text{CH}_3)=\text{CH}$, which is characteristic for a dihydrobenzoxanthone-type of flavone. Signals at 1.43, 5.73 and 6.71, as well as a doublet at δ 6.39, suggested the presence of a fused dimethylpyran ring in ring A. In addition, an aromatic singlet at 6.16 was observed for the remaining hydrogen of a pentasubstituted benzene ring (ring B).

From these structural units, structure 1 could be formulated. In the ^{13}C -NMR spectrum, also showed signal for carbonyl at δ 179.6 ppm. The chemical shift at δ 144.0 ppm showed signal for quarternary carbon. The chemical shift at δ 76.9 ppm showed signal for chromene, reinforced by chemical shift at δ 94.2; 116.7; 129.2 and 27.5 ppm. The chemical shift at δ 27.5 ppm is a typical shift of methyl chromene. Based on ^1H -NMR and ^{13}C -NMR data, compound 1 has the similar chemical shift with that of *Artoindonesianin A-3* (Syah, 2006).

4. Conclusion

A new prenylated flavone, *artoindonesianin A-3*, (melting point of 145-147°C) was isolated from *Artocarpus elasticus* collected from Alor Island of Indonesia.

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