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Isolation and Structural Elucidation of New Xanthone from Rot Bark of *Cratoxylum sumatranum*

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Abstract: Sumatranaxanthone A (1), a new xanthone, was isolated from the rot bark of *Cratoxylum sumatranum*. The structures were established based on spectroscopic studies, notably of the 1D and 2D NMR spectra. Preliminary results showed that 1 has antioxidative activity with inhibition 81% to fit with ascorbat acid standard.

Key words: Sumatranaxanthone · *Cratoxylum sumatranum* · Antioxidative activity

INTRODUCTION

Plants of the genus *Cratoxylum* are intriguing targets for phytochemical investigation not only because of their great structural variability but also because of the diverse biological activities of their secondary metabolites, among which are xanthones, benzophenones, flavonoids and terpenoids [1]. *C. sumatranum* is a high submountain dweller tree widely distributed in the West Province of Indonesia [2], where it is used as a medicinal plant against infections like dysentery, cold and toothache.

In this paper, we described the isolation and structural elucidation of a new xanthone derivative named sumatranaxanthone A (1) from the ethyl acetate extract of the rot bark of *C. sumatranum*. These structures of the compound were determined based on their NMR 1D and 2D data. We also reported antioxidant activity of xanthone isolated from *C. sumatranum*. The antioxidant activity was determined by DPPH method [3].

MATERIALS AND METHODS

Plant Material: The rot bark of *C. sumatranum* was collected in april 2008 in the Forest located in the West Province of Indonesia. A specimen was identified and maintained at the Herbarium Bogoriense, Bogor, Indonesia.

General Experimental Procedures: IR spectra was measured with Shimadzu 8300 FTIR. Melting point was determined on a micromelting point apparatus. ^{1}H and ^{13}C NMR spectra were recorded with Jeol JNM A-5000 spectrometers, operating at 600.0 MHz (^{1}H) and 150.0 MHz (^{13}C) using residual and deuterated solvent peaks as internal standards. Vacuum liquid chromatography (VLC) was carried out using Si-gel Merck 60 GF_{254} (230–400 mesh), column chromatography using Si-gel Merck 60 (200–400 mesh) and TLC analysis on precoated Si gel plates Si-gel Merck Kieselgel 60 GF_{254} 0.25 mm, 20 × 20 cm. The antioxydant activity was determined by DPPH free radical scavenging test (1,1-dipheniyl-2-picrylhydrazyl) method.

Extraction and Isolation: The dried and milled rot bark *C. sumatranum* (3 kg) was extracted with hexane, ethyl acetate and methanol respectively for 5 days at room temperature three times. Evaporation of the combined hexane, ethyl acetate and methanol extracts to dryness in vacuo, gave a brown residu 45 g, 60 g and 50 g respectively. A portion (25 g) of the ethyl acetate extract was subjected to vacum liquid chromatography eluted with a gradient system 90-20 % hexane-EtOAc to afford 4 fractions. Fraction 1 (1 g, resolved in ethyl acetate) was further purified in column chromatography eluted with gradient system 100-60% hexane - EtOAc to afford 4 subfraction. After purification, subfraction 2 was recrystalized to yield 1 (7 mg).

RESULTS AND DISCUSSION

Compound 1 was obtained as a yellow crystal, mp 220-222°C; IR (KBr) $\tilde{o}_{maks}$ 3352 cm^{-1} (OH), 1643 cm^{-1} (carbonyl), 1604, 1454 and 1400 cm^{-1} (benzene) and 1112 cm^{-1} (ether) ; ^{1}H NMR (chloroform- d_1, 500 MHz)

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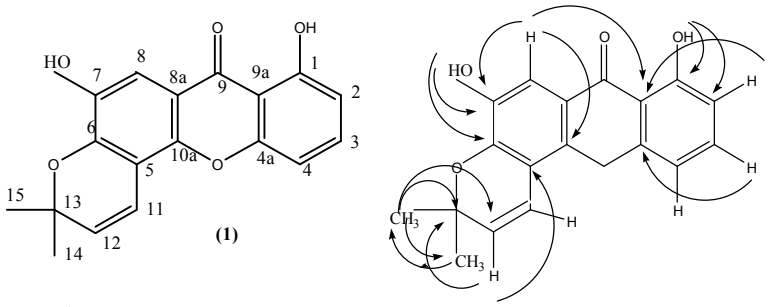

Fig. 1: HMBC of Compound 1

Table 1: ^{1}H, ^{13}C NMR and HMBC Spectra Data of compound 1.

Position	^{1}H NMR	^{13}C NMR	HMBC (carbon)
1		161,93	
1-OH	12.84 (1H,s)		C-1,C-2, C-9a
2	6.76 (1H,dd, 8.5, 1.2)	110,41	C-9[a],
3	7,53 (1H,t,8.5)	136.07	C-1, C-4[a]
4	6,90 (1H,dd, 8.5, 1.2)	106,87	
4[a]		156,27	
5		109.42	
6		146.67	
7		146,91	
7-OH	5.58 (1H,s)		
8	7,61 (1H,s)	108.87	C-6, C-7, C-9, C-10a
8-a		114.3	
9		181.47	
10[a]		142.25	
11	6.93 (1H, d, 10)	115.56	C-6, C- 7, C-13
12	5,76 (1H,d,10)	130.06	C-5, C-13
13		79.71	
14	1,56	28,46	C-12, C-13, C-15
15	1,56	26,46	C-12,C-13, C-14

δ_H ppm and ^{13}C NMR (chloroform- d_1, 125 MHz) δ_C ppm. The IR spectrum of compound 1 exhibited absorptions for hydroxyl (3352 cm^{-1}), carbonyl (1643 cm^{-1}), aromatic (1604, 1454 and 1400 cm^{-1}) and ether (1112 cm^{-1}). The ^{1}H NMR Spectrum (Table 1) showed a sharp single of a chelated hydroxy proton (1-OH) at δ 12.84, a broad singlet signal of a phenolic proton at δ 5.58, an proton aromatic at δ 7.61 (1H, s).and typical signals of a dimethylchromene ring 6.73, 5.59 (1H each, d, 10.4 Hz) and 1.48 (6H, s), in addition to the presence of a 1,2,3-trisubstituted benzene signals at 7.53 (1H, t, 8.5), 6.90 and 6.76 (1H each, dd, 8.5 and 1.2 Hz). From HMQC spectra showed the aromatic proton at δ_H 6.76 connection to a carbon signal at δ_C110.41 ppm, δ_H 7.53 connection to a carbon δ_C 136.07 and 6.90 connection to a carbon δ_C 106.87. In the HMQC also showed the proton methine at δ_H 6.93 and 5.76 connection to carbon signal at δ_C 115.56 and 130.06 respectively. In the HMBC data, the chelated hydroxy proton (1-OH) showed a cross peak with a aromatic carbon at δ_C 161.93 and aromatic methine carbon at 110.4 which correlated to an aromatic proton of a1,2,3-risubstituted benzene ring at δ_H 6.76 (1H, dd, 8.5, 1.2) in the HMQC spectrum. These result indicated that this aromatic proton was located at C-2. Two aromatic protons at δ_H 7.53 (1H, t, 8.5) and δ_H 6,90 (1H,dd, 8.5, 1.2) where then attributed to H-3 and H-4, respectively. In the HMBC spectrum showed cross peak between H-3 with C-4a (δ_C 156.27). The linkage of the cromene ring at C-5 (δ_C 109.42) and C-6 with an ether linkage at C-6 was established by HMBC data (Tabel 1), which showed cross peak H-12 (δ_H 5.76) with C-5. Therefore was determined as 1,7-dihydroxyy-13,13 – dimehylpyrano (11,12: 6,5) xanthone and after direct comparison with those of reported data the same as sumatranaxanthone A. The antioxidant activity was determined by DPPH (1,1-dipheniyl-2-picrylhydrazyl) method and compound 1 showed antioxidant activity with inhibition 81 % to fit with ascorbat acid standard.

CONCLUSION

In conclusion, we have reported new Xanthone from Rot Bark of*Cratoxylum sumatranum.* Preliminary results showed that 1 has antioxidative activity with inhibition 81% to fit with ascorbat acid standard.

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