24

Chemical Constituents Of Mitrella Kentii (Annonaceae)

Ainnul Hamidah Syahadah Azizan^{*} and A. Hamid A. Hadi

Department of Chemistry, Faculty of Science, 50603 Kuala Lumpur

Abstract: Mitrella kentii which belongs to the Annonaceae family is a tree-climbing liana found in the Malaysian Peninsula, Islands of Sumatra, Borneo, and New Guinea. This species was studied in 1972 for their alkaloids, 1997 and 2012 for their non-alkaloid constituents. In this study, chalcones, Desmosdumotin C (1) and their tautomer, 2-cinnamoyl-3-hydroxy-5-methoxy-4,6,6-trimethylcyclohexa-2,4-dienone (2), flavanone, 7-hydroxy-5,6-dimethoxy-2-phenylchroman-4-one (3), oxoaporphine alkaloids, Liriodenine (4) and Atherospermidine (5) and also terpenoid, β -Sitostenone (6) have been isolated from this species. All these compounds were isolated for the first time from Mitrella kentii except Liriodenine (4). The isolated compounds were elucidated using spectroscopic techniques such as UV, IR,1D and 2D NMR and mass spectroscopy and by comparison their spectral data with those previously reported in the literatures. Desmosdumotin C (1) showed the anti-ulcer activity.

Keywords: Annonaceae, Alkaloid, Anti-ulcer activity, Desmosdumotin C, Mitrella kentii.

1. INTRODUCTION

Mitrella kentii from the family Annonaceae is a treeclimbing liana belonging to the custard apple family found in the Malaysian Peninsula and in the islands of Sumatra, Borneo, and New Guinea [1, 2]. Its synonyms are Melodorum pisocarpum and M. elegans, while the common (Indonesian) name is 'kiawi'. The plant is found in the tropics, especially in the Asia-Pacific regions. It is consumed in the form of a root decoction to treat fever in Malaysia. From the previous chemical studies on the steam bark of this species, in 1972, they are found oxoaporphine and isoquinoline types of alkaloid [1] and in 1997 for non-alkaloid constituents [2]. In our continuous research for biologically active compounds from the Malaysian flora, we started on the hexane and dicholoromethane extracts of the stem bark of this plant for phytochemical investigations. This study led to the isolation of six known compounds; Desmosdumotin C (1) and its tautomer (2), 7-hydroxy-5,6-dimethoxy-2-phenylchroman-4-one (3), Liriodenine (4), Atherospermidine (5) and β -Sitostenone (6).

2. RESULTS AND DISCUSSION

Desmosdumotin C (1) was previously isolated from the roots of *Desmos dumosus* [3] and *Campomanesia lineatifoli*⁴ and it was isolated for the first time from Mitrella kentii. Desmosdumotin C (1) was a novel chalcone and it structure has been established by X-ray crystallography together with 1D and 2D NMR spectroscopy.

Desmosdumotin C (1), yellow needle crystals, has the molecular formula C19H20O4 was established by highresolution mass measurement (HREIMS) of the molecular ion peak $[M+H]^+$ at m/z 313.1435 and elemental analysis

E-mail: ainnul_azizan@yahoo.com

data. Its IR spectrum showed the absorption peaks at 1657 cm⁻¹ indicating the presence of conjugated carbonyl group, the alkene group absorbed at 1624 cm⁻¹ and aromatic rings at 1576 and 1514 cm⁻¹. The ¹H NMR spectrum showed signals for a methoxyl group at δ 3.93 as a singlet, olefinic methyl group resonated at δ 2.02 as a singlet and two geminal methyl groups appeared at δ 1.36 as a singlet with six protons. The aromatic protons were resonated at δ 7.66-7.37 as multiplet and two trans-oriented olefinic protons were resonated at δ 8.32 as a doublet with J value 16.0 Hz and δ 7.92 as a doublet with J value 16.0 Hz which were assigned for C2'-H and C3'-H respectively. The ¹³C-NMR spectrum showed the presence of nineteen signals which belong to eight quaternary carbons, seven methine group, three methyls and one methoxyl group. From the foregoing results, the compound was identified as Desmosdumotin C. In addition, the chemical structure was confirmed by the X-ray crystallographic structure analysis as seen in Fig. (2) and Fig. (3) showed the HMBC, which is the correlations between hydrogen and carbon.

3. EXPERIMENTAL SECTION

3.1 General Experimental Procedure

The ¹H- NMR and ¹³C-NMR experiments were performed on a JOEL 400 MHz spectrometer, a Bruker 400 MHz and 600MHz, respectively. The 2-D NMR experiments were performed on the JOEL and Bruker spectrometer using appropriate pulse sequence programs. The IR spectra were obtained on a Perkin Elmer 1600 Double-Beam recording spectrometer. HREIMS were determined on Agilent Technologies 6530 Accurate-Mass Q-TOF LC/MS. The UV spectra were recorded on a Shimadzu UV-160A ultravioletvisible spectrometer.

3.2. Plant Material

The bark of Mitrella kentii (B1.) Miq. was collected in Mersing, Johor with a voucher specimen (KL 4139) and was

^{*}Address correspondence to this author at the Department of Chemistry, Faculty of Science, 50603 Kuala Lumpur;

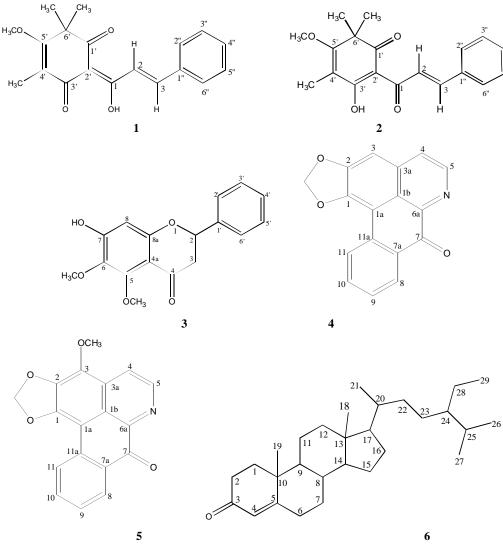


Fig. (1). Chemical constituents of *M. kentii*.

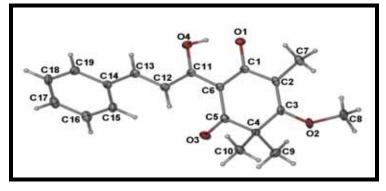


Fig. (2). X-ray crystallographic.

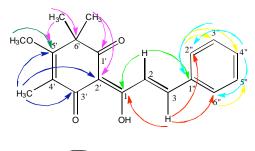
deposited at the Herbarium of Department of Chemistry, University of Malaya, Kuala Lumpur, Malaysia.

3.3. Extraction and Isolation

The dried and ground bark of *M. kentii* (1.0 kg) was first extracted with hexane followed by CH_2Cl_2 and methanol. The hexane, CH_2Cl_2 and methanol extracts were evaporated to dryness to give crude hexane, CH_2Cl_2 and methanol extract, respectively. These extracts were subjected to

column chromatography using silica gel 60 and preparative TLC on Si gels. Two compounds were isolated from the hexane extract: Desmosdumotin C (1), its tautomer (2) and β -Sitostenone (6). 7-hydroxy-5,6-dimethoxy-2-phenylchroman-4-one (3), Liriodenine (4) and Atherospermidine (5) were isolated from the CH₂Cl₂ extract.

Desmosdumotin C (1): $C_{19}H_{20}O_4$, isolated as a yellow needle crystals from a n-hexane – CH_2Cl_2 mixture, m.p.: 93-



H to Carbon Correlation

Fig. (3). Selected 2D NMR correlations of Desmosdumotin C (1).

94 °C; UV $\lambda_{methanol}$: 380, 242, 225 nm; IR_{max} (cm⁻¹, NaCl disc): 3401, 1657, 1624, 1577, 1513, 1426, 1371, 1243, 1153, 1122, 977, 944; EIC-MS m/z [M+H]⁺ (%): 312.140729 (calc. 312.3646 for C₁₉H₂₀O₄); ¹H NMR (CDCl₃, TMS) δ (ppm): 8.3(1H, d, *J*= 16Hz), 7.9(1H, d, *J*=16Hz), 7.7(2H, m, Ar-2",6"-H), 7.4(3H, s, Ar-3", 4", 5"-H), 3.9(3H, s, OCH₃), 2.0(3H, s, Ar-CH₃) 1.7(6H, s, CH₃x2). ¹³C NMR (CDCl₃, TMS) δ (ppm) : 198.1(C-1'), 192.5(C-3'), 187.3(C-1), 176.7(C-5'), 123.3(C-2), 144.9(C-3), 135.3 (C-1"), 130.7(C-3",C-4",C-5"), 128.9(C-2",C-6"), 113.7(C-2'), 106.7(C-4'), 62.2(C-5'-OCH₃), 50.5(C-6'), 24.4(C6'-CH₃x2),

Received: May 29, 2013

Revised: September 24, 2013

Accepted: October 10, 2013

© Azizan and Hadi; Licensee Bentham Open.

This is an open access article licensed under the terms of the Creative Commons Attribution Non-Commercial License (http://creativecommons.org/licenses/by-nc/3.0/), which permits unrestricted, non-commercial use, distribution and reproduction in any medium, provided the work is properly cited.

 $9.9(C4'-CH_3)$. The compound was identified by comparison of their spectroscopic data with literature values.

CONFLICT OF INTEREST

The authors confirm that this article content has no conflicts of interest.

ACKNOWLEDGEMENTS

Financial support by HIR-MOHE F000009-21001 is greatly appreciated.

REFERENCES

- Ellis, J.; Gellert, E.; Summons, R. The alkaloids of *Mittella kentii* (Annonaceae). Aus. J. Chem., 1972, 25 (12), 2735-2736.
- [2] Benosman, A.; Oger, J.-M.; Richomme, P.; Bruneton, J.; Roussakis, C.; BöschT, S.; Ito, K.; Ichino, K.; Hadi, A.H.A. New terpenylated dihydrochalcone derivatives isolated from *Mitrella kentii. J. Nat. Prod.*, **1997**, *60*(9), 921-924.
- [3] Wu, J.H.; McPhail, A.T.; Bastow, K.F.; Shiraki, H.; Ito, J.; Lee, K.H.; Desmosdumotin, C. A novel cytotoxic principle from *Desmos dumosus. Tetrahedron Lett.*, 2002, 43(8), 1391-1393.
- [4] Bonilla, A.; Duque, C.; Garzón, C.; Takaishi, Y.; Yamaguchi, K.; Hara, N.; Fujimoto, Y. Champanones, yellow pigments from the seeds of champa (*Campomanesia lineatifolia*). *Phytochemistry* 2005, 66(14), 1736-1740.