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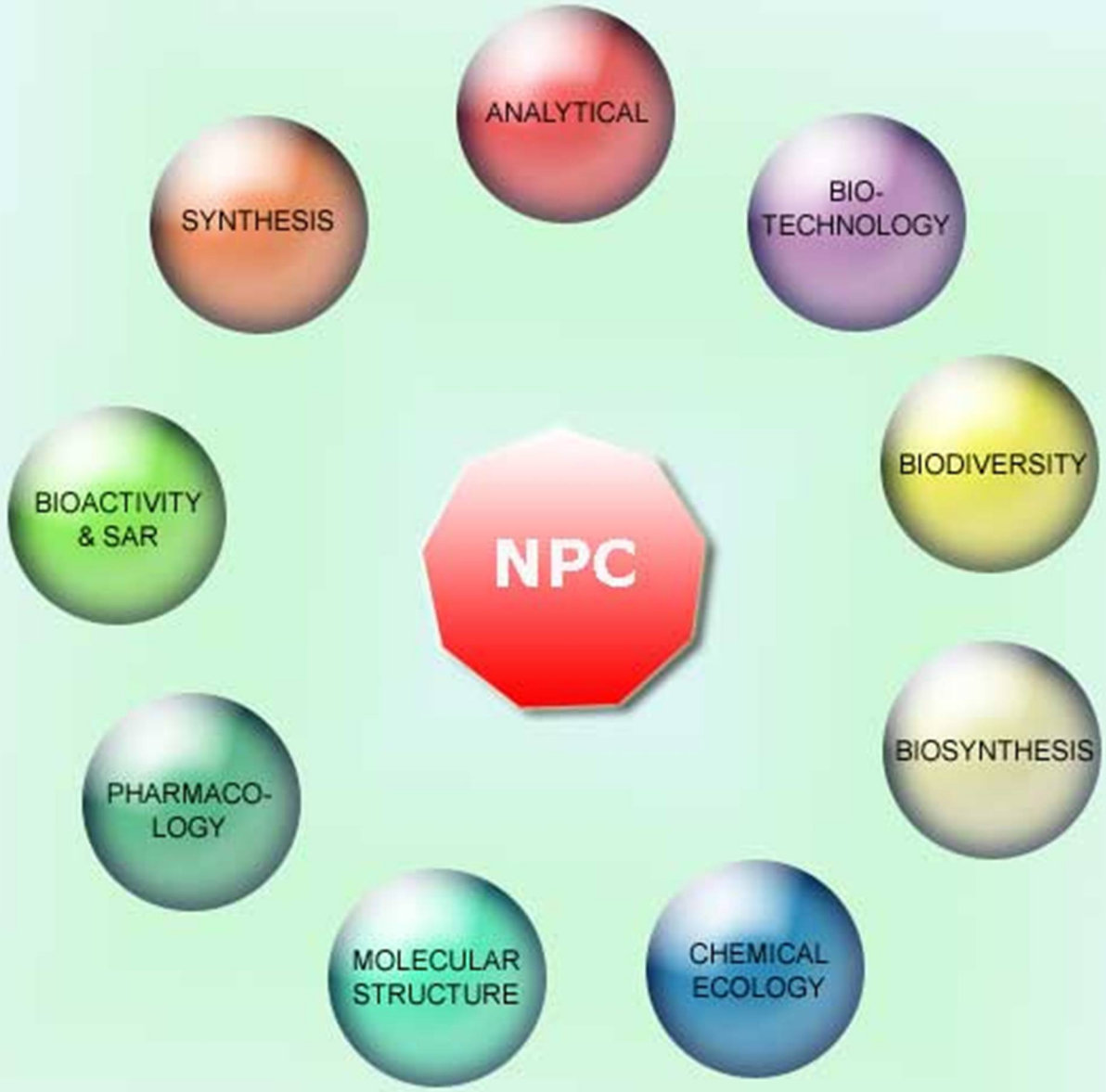

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Flavonoids and Triterpenes from the leaves of *Artocarpus fulvicortex*

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Five flavonoids, 5-hydroxy-(6:7,3′:4′)-di(2,2-dimethylpyrano)flavone **1**, carpachromene **2**, cycloartocarpesin **3**, norartocarpetin **4** and 2′-hydroxy-4,4′,6′-trimethoxychalcone **5**, along with three triterpenes, friedelin **6**, lupeol **7** and β-sitosterol **8** were isolated for the first time from the leaves of *Artocarpus fulvicortex* F.M. Jarrett. The structures of these compounds were established by analysis of their spectroscopic (1D and 2D NMR) and spectrometric (MS) data, as well as by comparison of these with those reported in the literature.

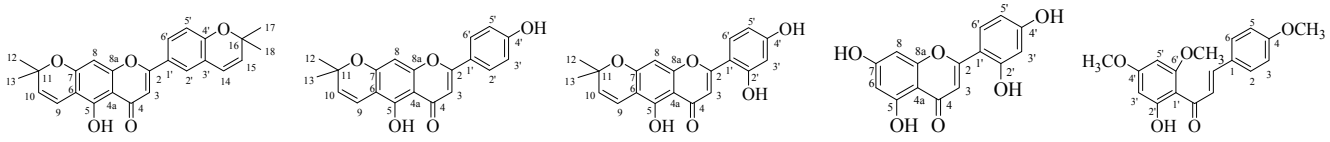
Keywords: *Artocarpus fulvicortex*, Moraceae, Flavonoids, Triterpenes.

The genus *Artocarpus* of the family Moraceae consists of approximately 50 species of which 11 are known to produce edible fruits and most species are indigenous to the tropical rain forests of Malaysia, Indonesia and Philippines [1a]. *Artocarpus fulvicortex* F.M. Jarret is one of Malaysia's rare *Artocarpus* species and can be found only in the rainforests of Besut situated at the East Coast of Peninsular Malaysia. This plant is locally known as 'keledang tampang gajah'. It is a medium-sized tree with orange brown or reddish brown bark [1b]. Previous phytochemical work on the genus *Artocarpus* has resulted in the isolation of numerous phenolic compounds especially flavonoids [2a-2d]. Flavonoids isolated from *Artocarpus* species showed interesting biological activities including, antiplatelet [2e, 2f], antimicrobial [2g], antioxidant [2h], antiinflammatory [2i] and also exhibited cytotoxic effects [2j]. In continuing research on Malaysian *Artocarpus* plants, we investigated the leaves of *A. fulvicortex* F.M. Jarrett. Here, we wish to report for the first time the chemical constituents of this species.

The powdered leaves of *A. fulvicortex* were sequentially extracted with petroleum ether (PE), CH_2Cl_2, EtOAc and MeOH at room temperature. The MeOH crude extract was further suspended in water before partitioned with CH_2Cl_2 and EtOAc to yield CH_2Cl_2-soluble extract and EtOAc-soluble extract. The chromatographic separations using vacuum liquid chromatography (VLC) and column chromatography (CC) on silica gel of the PE extract, followed by recrystallization afforded **1**, **5**, **6** and **7**. Purification by VLC and CC of the CH_2Cl_2 extract furnished **2** and **8**. Purification using VLC and repeated CC of EtOAc and EtOAc-soluble extracts yielded **3** and **4**. The structures of **1-8** were elucidated by comparison of the spectroscopic data with respective literature [2a-7c]. Compound **1** was found to be a new pyranoflavone.

Compound **1** (2.0 mg, 0.01%) was obtained as a pale yellow solid with m.p. 215-217ºC. The TLC of **1** gave a yellow spot after spraying with vanillin sulphuric acid reagent suggestive of a flavone-type structure. The molecular formula was determined to be $C_{25}H_{22}O_5$ from its HREIMS. The ^{1}H NMR of **1** showed signals at δ 6.41 (1H, d, *J* = 10.0 Hz, H-14), δ 5.73 (1H, d, *J* = 10.0 Hz, H-15) and δ 1.49 (6H, s, H-17 and H-18); δ 6.74 (1H, d, *J* = 10.0 Hz, H-9), δ 5.64 (1H, d, *J* = 10.0 Hz, H-10) and δ 1.48 (6H, s, H-12 and H-13), characteristic of two sets of 2,2-dimethylpyrano moieties, as well as three signals at δ 7.65 (1H, dd, *J* = 8.8 and 2.4 Hz, H-6′), δ 7.49 (1H, d, *J* = 2.4 Hz, H-2′) and δ 6.89 (1H, d, *J* = 8.8 Hz, H-5′) corresponding to an ABX spin system of ring B. Other signals observed were at δ 13.14 (OH, s), characteristic of a chelated hydroxyl group, as well as at δ 6.53 (1H, s, H-3) and δ 6.41 (1H, s, H-8) for isolated aromatic protons. In the ^{13}C NMR spectrum, a resonance for a carbonyl group at δ 182.4 was observed together with resonances for four methyl groups (δ 28.2, δ 28.3, δ 29.6 and δ 30.9). NMR data comparison with carpachromene (**2**) [3a,b] and other pyranoflavonoids [3c] was carried out to confirm the position of both 2,2-dimethylpyrano moieties in the structure. Thus, compound **1** was deduced as 5-hydroxy-(6:7,3′:4′)-di(2,2-dimethylpyrano)flavones. To the best of our knowledge, the isolation of **1** from *A. fulvicortex* or other plant has not been reported elsewhere.

This is also the first report on the presence of 2′-hydroxy-4,4′,6′-trimethoxychalcone **5** in *Artocarpus* species. Previously, **5** has been encountered from *Goniothalamus gardneri* (Annonaceae) [4a] and *Andrographis lineate* (Acanthaceae) [4b]. Carpachromene **2** has been previously isolated from *A. bracteata* [2b] and *A. heterophyllus* [3a] while cycloartocarpesin **3** has been previously identified from *A. heterophyllus* [3a] and *A. elasticus* [5a]. The occurrence of **3** was also reported in *Maclura pomifera*, one of the Moraceae species [5b]. Norartocarpetin **4** was previously isolated from *A. champeden* [6a], *A. scortechinii*, *A. kemando*, and *A. gomezianus* [2b], *A. heterophyllus* [6b], and *A. dadah* [6c]. Friedelin **6**, lupeol **7** and β-sitosterol **8** are commonly found in plants [7a-7c].



(1) **(2)** **(3)** **(4)** **(5)**

Experimental

Plant material: The leaves of *Artocarpus fulvicortex* J.M. Jarrett were collected from Besut, Terengganu in September 2007. A voucher specimen (HTBP 962) has been deposited at Putrajaya Botanical Garden Herbarium, Malaysia.

Isolation procedure: Sequential extraction of the dried powdered leaves (1.5 kg) of *A. fulvicortex* at room temperature using different polarity of organic solvents for 48 h each afforded the PE extract (16.1 g, 1.1%), the CH_2Cl_2 extract (18.0 g, 1.2%), the EtOAc extract (17.9 g, 1.2%), and the MeOH extract (78.5 g, 4.2%). The MeOH extract was further suspended in water, then sequentially partitioned with CH_2Cl_2 and then EtOAc to yield the CH_2Cl_2-soluble extract (AFLMD) (1.1 g, 1.4%) and the EtOAc-soluble extract (AFLME) (1.2 g, 1.5%). Fractionation of the leaves PE extract (15.0 g) by silica gel VLC afforded twenty five fractions of 100 mL each. Fractions with a similar TLC profile were combined to give six major fractions AFLP 1 - AFLP 6. Repeated CC of AFLP 3 (1.7 g), followed by PLC with hexane/Et_2O (7:3) as the solvent system gave **1** (2.0 mg, 0.01%) as a pale yellow solid. Successive purification of AFLP 4 (3.4 g) using silica gel CC yielded **6** (115.8 mg, 0.72%) as white needles; **7** (23.6 mg, 0.15%) as a white powder and **5** (9.3 mg, 0.06%) as a yellow solid. Fractionation of the leaves CH_2Cl_2 extract (12.0 g) by VLC yielded seven major fractions. Purification of fraction 5 (1.6 g) by silica gel CC gave 446 fractions. Fractions 187-264 were combined and washed with hexane to afford **8** (112.2 mg, 0.62%) as white crystalline needles. Purification of fraction 6 (2.8 g) by silica gel CC yielded carpachromene **2** (99.2 mg, 0.55%) as pale yellow needles. Fractionation of the EtOAc extract (10.0 g) by silica gel VLC afforded twenty fractions which were combined to yield seven major fractions (AFLE 1 - AFLE 7). Purification of AFLE 3 (0.9 g) by silica gel CC afforded **3** (21.2 mg, 0.12%) as pale yellow needles. Purification of the EtOAc-soluble fraction of the MeOH extract (1.0 g) using silica gel CC and hexane/EtOAc (7:3) as the solvent yielded **5** (13.6 mg, 0.14%) as a yellow solid.

5-Hydroxy-(6:7,3′:4′)-di(2,2-dimethylpyrano)flavone (**1**)
MP: 215-217ºC.
IR (KBr): 3441, 2923, 1655, 1606, 1127 cm^{-1}.
^{1}H NMR (400 MHz, $CDCl_3$): 1.49 (6H, s, H-17 and H-18), 1.48 (6H, s, H-12 and H-13), 5.64 (1H, d, *J* = 10.0 Hz, H-10), 5.73 (1H, d, *J* = 10.0Hz, H-15), 6.41 (1H, d, *J* = 10.0 Hz, H-14), 6.41 (1H, s, H-8), 6.53 (1H, s, H-3), 6.74 (1H, d, *J* = 10.0 Hz, H-9), 6.89 (1H, d, *J* = 8.8 Hz, H-5'), 7.49 (1H, d, *J* = 2.4 Hz, H-2'), 7.65 (1H, dd, *J* = 8.8 Hz, 2.4 Hz, H-6'), 13.14 (1H, s, 5-OH).
^{13}C NMR ($CDCl_3$, 100 MHz): δ 28.2 (CH_3), 28.3 (CH_3), 29.6 (CH_3), 30.9 (CH_3), 72.6 (C), 77.9 (C), 94.9 (CH), 104.0 (CH), 105.4 (C), 105.5 (C), 109.3 (C), 115.5 (CH), 116.9 (CH), 121.5 (CH), 123.5 (C), 124.3 (CH), 127.4 (CH), 128.0 (CH), 131.6 (CH), 157.4 (C), 156.4 (C), 159.7 (C), 161.5 (C), 163.8 (C), 182.4 (C=O).
MS (EI, 70 eV): *m/z* (%) 402 (27) [M^+], 387 (100), 219 (29), 203 (13), 186 (32), 131 (12); HREIMS: *m/z* [M+] calcd. for $C_{25}H_{22}O_5$: 402.14574; found 402.14618.

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Conservation and Multiplication of Encapsulated Micro Shoots of *Rauvolfia vomitoria* - an Endangered Medicinal Tree: ISSR and RAPD based Evaluation of Genetic Fidelity of Converted Plantlets
Shakti Mehrotra, Liaq Ur Rahman, Jahnvi Mishra and Arun K. Kukreja **1647**

Bioactivities of Volatile Components from Nepalese *Artemisia* Species
Prabodh Satyal, Prajwal Paudel, Ananad Kafle, Suraj K. Pokharel, Bimala Lamichhane, Noura S. Dosoky, Debra M. Moriarity and William N. Setzer **1651**

Chemical Compositions, Antioxidant and Antimicrobial Activity of the Essential Oils of *Piper officinarum* (Piperaceae)
Wan Mohd Nuzul Hakimi Wan Salleh, Farediah Ahmad, Khong Heng Yen and Hasnah Mohd Sirat **1659**

Chemical Composition, Antimicrobial and Antioxidant Properties of the Volatile Oil and Methanol Extract of *Xenophyllum poposum*
Ana M. González, María I. Tracanna, Sara M. Amani, Carola Schuff, Miguel J. Poch, Horacio Bach and César A. N. Catalán **1663**

Phytochemical and Micromorphological Traits of Endemic *Micromeria pseudocroatica* (Lamiaceae)
Dario Kremer, Ivna Dragojević Müller, Edith Stabentheiner, Dubravka Vitali, Martina Kopričanec, Mirko Ruščić, Ivan Kosalec, Nada Bezić and Valerija Dunkić **1667**

Composition and Variability of the Essential Oil of *Salvia nemorosa* (Lamiaceae) from the Vienna Area of Austria
Remigius Chizzola **1671**

Essential oil Composition of *Ficus benjamina* (Moraceae) and *Irvingia barteri* (Irvingiaceae)
Isiaka A. Ogunwande, Razaq Jimoh, Adedoyin A. Ajetunmobi, Nudewhenu O. Avoseh and Guido Flamini **1673**

Review/Account

Cytotoxic Agents of the Crinane Series of Amaryllidaceae Alkaloids
Jerald J. Nair, Jaume Bastida, Francesc Viladomat and Johannes van Staden **1677**

Natural Product Communications
2012
Volume 7, Number 12

Contents

Original Paper ***Page***

A Comparative Study of the Antioxidant/Prooxidant Effects of Carvacrol and Thymol at Various Concentrations on Membrane and DNA of Parental and Drug Resistant H1299 Cells
Aysun Ozkan and Ayse Erdogan **1557**

New Secoiridoid Glycosides from the Buds of *Lonicera macranthoides*
Jiang Liu, Jing Zhang, Feng Wang and Xingfu Chen **1561**

Chemical Constituents from the Aerial Parts of *Gynura bicolor*
Jian Chen, Sven Mangelinckx, An Adams, Wei-lin Li, Zheng-tao Wang and Norbert De Kimpe **1563**

Chemical Constituents of *Ligularia nelumbifolia* and *L. subspicata* Hybrid Collected in Shangrila County, Yunnan Province of China
Ryo Hanai, Hiroka Yamada, Yurika Suzuki, Hajime Nagano, Takayuki Kawahara, Jiao-Jun Yu, Xun Gong and Chiaki Kuroda **1565**

A Practical, Enantiospecific Synthesis of (*S*)-*trans*-γ-Monocyclofarnesol
Stefano Serra **1569**

ACAT Inhibitory Activity of Exudates from *Calocedrus macrolepis* var. *formosana*
Yu-Hsin Hsieh, Kuan-Jung Chen, Shih-Chang Chien, Wen-Ling Cheng, Jun-Hong Xiao and Sheng-Yang Wang **1573**

Cucurbitane-Type Triterpenoids from the Fruit Pulp of *Momordica charantia*
Yun-Wen Liao, Chiy-Rong Chen, Yueh-Hsiung Kuo, Jue-Liang Hsu, Wen-Ling Shih, Hsueh-Ling Cheng, Tzou-Chi Huang and Chi-I Chang **1575**

Two Novel Phenethylamine Alkaloids from *Streptomyces* sp. YIM10049
Xueqiong Yang, Guangwei He, Lixing Zhao, Yabin Yang, Yun Liu, Lihua Xu and Zhongtao Ding **1579**

Alkaloids from *Cinnamomum philippinense*
Hsing-Tan Li, Wei-Jen Li, Hui-Ming Wu and Chung-Yi Chen **1581**

Conversional Synthesis of Heteratisine
Ling Wang, Qi-Feng Chen and Feng-Peng Wang **1583**

Flavonoids and Triterpenes from the leaves of *Artocarpus fulvicortex*
Shajarahtunnur Jamil, Muhammad Taher, Hasnah M. Sirat and Nur Azlin Othman **1587**

Dihydrospinochalcone-A and Epi-flemistrictin-B, Natural Isocordoin Derivatives from the Root Extract of *Lonchocarpus xuul*
Fabiola Escalante-Erosa, Brenda González-Morales, Ramiro F. Quijano-Quiñones, Gumersindo Miron-López and Luis M. Peña-Rodríguez **1589**

Phenolic Compounds, Including Novel *C*-glycosylflavone, from the Flowers of the Tall Bearded Iris Cultivar 'Victoria Falls'
Takayuki Mizuno, Tsutomu Yabuya, Nobuhiro Sasaki and Tsukasa Iwashina **1591**

Two New Biisoflavonoids from the Roots of *Daphne oleoides*
Shazia Yasmeen, Muhammad Aijaz Anwar, Sadia Ferheen, Nighat Afza, Abdul Malik and Lubna Iqbal **1595**

Biflavonoids from the Unripe Fruits of *Clusia paralicola* and their Antioxidant Activity
Rafaela Ferreira Oliveira, Celso Amorim Camara, Maria de Fátima Agra and Tania Maria Sarmento Silva **1597**

Ochnaflavone and Ochnaflavone 7-*O*-methyl Ether two Antibacterial Biflavonoids from *Ochna pretoriensis* (Ochnaceae)
Tshepiso J. Makhafola, Babatunde B. Samuel, Esameldin E. Elgorashi and Jacobus N. Eloff **1601**

Factors Affecting the Separation and Bioactivity of Red Clover (*Trifolium pratense*) Extracts Assayed against *Clostridium sticklandii*, a Ruminal Hyper Ammonia-producing Bacterium
Isabelle A. Kagan and Michael D. Flythe **1605**

Exploitation of the Antioxidant Potential of *Geranium macrorrhizum* (Geraniaceae): Hepatoprotective and Antimicrobial Activities
Niko S. Radulović, Milan B. Stojković, Snežana S. Mitić, Pavle J. Randjelović, Ivan R. Ilić, Nikola M. Stojanović and Zorica Z. Stojanović-Radić **1609**

Differential Expression of Benzophenone Synthase and Chalcone Synthase in *Hypericum sampsonii*
Lili Huang, Hong Wang, Hechun Ye, Zhigao Du, Yansheng Zhang, Ludger Beerhues and Benye Liu **1615**

Efficient Preparation of Hangekobokuto (Banxia-Houpo-Tang) Decoction by Adding Perilla Herb before Decoction is Finished
Megumi Sumino, Yuko Saito, Fumio Ikegami, Yoshiro Hirasaki and Takao Namiki **1619**

Anti-neutrophilic Inflammatory Secondary Metabolites from the Traditional Chinese Medicine, Tiankuizi
Chia-Lin Lee, Tsong-Long Hwang, Chieh-Yu Peng, Chao-Jung Chen, Yuan-Shiun Chang and Yang-Chang Wu **1623**

DART MS Based Chemical Profiling for Therapeutic Potential of *Piper betle* Landraces
Vikas Bajpai, Renu Pandey, Mahendra Pal Singh Negi, Nikhil Kumar and Brijesh Kumar **1627**

Total Phenolic Content and Antioxidative Properties of Commercial Tinctures Obtained from Some Lamiaceae Plants
Adam Kowalczyk, Izabela Biskup and Izabela Fecka **1631**

Synthesis, Antifungal Activity and Structure-Activity Relationships of Vanillin oxime-*N*-*O*-alkanoates
Vivek Ahluwalia, Nandini Garg, Birendra Kumar, Suresh Walia and Om P. Sati **1635**

Cinnamic Acid Derivatives Acting Against *Aspergillus* Fungi. *Taq* Polymerase I a Potential Molecular Target
Lucas J. Gutierrez, Maria L. Mascotti, Marcela Kurina-Sanz, Carlos R. Pungitore, Ricardo D. Enriz and Fernando A. Giannini **1639**

Insecticidal Effects of Acetogenins from *Rollinia occidentalis* Seed Extract
Diego Tolosa, Olga Álvarez Colom, Alicia Bardón and Adriana Neske **1645**

Continued inside backcover