

The Flavonoids of *Orophea polycarpa* A. DC. (Annonaceae)

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ABSTRAK

Kajian ke atas bahagian neutral *Orophea polycarpa* A. DC. telah menghasilkan dua flavonoid yang dikenali sebagai 2'-hidroksi-4,4',6'-trimetoksikalkon dan 5,7,4'-trimetoksiflavanon.

ABSTRACT

Investigation on the neutral fraction of the leaves of *Orophea polycarpa* A. DC. led to the isolation of two flavonoids identified as 2'-hydroxy-4,4',6'-trimethoxychalcone and 5,7,4'-trimethoxyflavanone.

Keywords: chalcone; flavanone; *Orophea polycarpa*; ¹H nuclear magnetic resonance spectrometry; ¹³C nuclear magnetic resonance spectrometry; infra-red & uv-visible spectrophotometry; flavonoids.

INTRODUCTION

Continuing investigations on the medicinal plants of Peninsular Malaysia, the chemical constituents, including the isolation of flavonoids, of *Orophea polycarpa* A. DC. (Annonaceae) were studied. *Orophea polycarpa* has not previously been investigated chemically but two aristolactams, enterocarpam I and enterocarpam II have recently been reported from *O. enterocarpam* Maingay ex Hk.f. & Thoms (Mahmood *et al.* 1986). *Orophea polycarpa* is a small tree, approximately 8 m tall which grows in the north of Peninsular Malaysia and is reported to have bechic and sudorific properties (Perry and Metzger 1980).

MATERIALS AND METHODS

General

The leaves of this species were collected from the foot of a limestone hill in Langkawi Island, off the northwest coast of Peninsular Malaysia. Voucher

specimen No: 4718 was deposited in the herbarium of the Biology Department, Universiti Pertanian Malaysia.

Melting points were determined on a Kofler hot stage and were uncorrected. Infrared and ultraviolet spectra were recorded on Beckmann Acculab 3 and Hitachi 200-20 spectrophotometers, respectively. ^1H NMR and ^{13}C NMR were recorded on a Bruker CPX 300 instrument at 300 and 75.5 MHz, respectively. Mass spectra were obtained from a Hewlett-Packard 5970 Mass Selective Detector coupled to a Hewlett Packard 5890 gas chromatography fitted with an open split capillary interface and HP-1 (polymethylsiloxane, 0.17 μ ; 25 \times 0.32 mm i.d.) capillary column with helium carrier gas (2 ml/min. flow rate; 15 psi). Column and thin-layer chromatography utilized Merck 7734 and 7730 silica gel, respectively.

Extraction

The air-dried leaves (1.1 kg) were ground to powder and defatted with petroleum ether (2 \times 2 L). The combined methanol extracts were evaporated under reduced pressure to give 24.1 g of crude extract which was fractionated between chloroform and 2N aqueous sulphuric acid. The chloroform fraction was evaporated to give 5.0 g of dark green semi-solid which was subjected to column chromatography (5 cm i.d. \times 35 cm) using silica gel as adsorbent and chloroform:methanol mixture as eluent in increasing polarity manner.

The combined major fractions consisted of two major components which were separated on preparative thin-layer chromatography using silica gel and developed with 2% methanol in chloroform. The more polar compound-I (30 mg) which crystallized from CHCl_3 , was found to convert into the less polar compound-II (40 mg) on standing in N_2 atmosphere.

Compound-II, 5,7,4'-trimethoxyflavanone. Bright yellow crystals: m.p. 115-117 $^\circ\text{C}$ (lit; 123.5-124.5 $^\circ\text{C}$, Kaufmann and Lam 1967; 195 $^\circ\text{C}$, Mishra and Misra 1980). Spectral data were in accord with literature.

UV : Kaufmann and Lam 1967; Lam and Wrang 1975.
IR : Kaufmann and Lam 1967; Dutta and Som 1978.
 ^1H NMR : Kaufmann and Lam 1967; Dutta and Som 1978.
 ^{13}C NMR : Duddeck *et al.* 1978.
MS : Lam and Wrang 1975.

Compound-I, 2'-hydroxy-4,4',6'-trimethoxychalcone. Pale yellow crystals: m.p. 95-97 $^\circ$ (lit. 115 $^\circ\text{C}$, Tuntiwachwuttikul *et al.* 1987). Spectral data were in accord with literature.

UV : Hansel *et al.* 1963; Lam and Wrang 1975; Tuntiwachwuttikul *et al.* 1987.

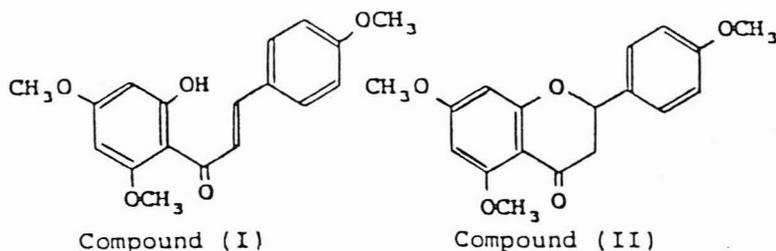
- IR : Hansel *et al.* 1973; Dutta and Som 1978; Tuntiwachwuttikul *et al.* 1987.
¹H NMR : Dutta and Som 1978; Tuntiwachwuttikul *et al.* 1987.
¹³C NMR : Duddeck *et al.* 1978.
 MS : Lam and Wrang 1975; Dutta and Som 1978; Tuntiwachwuttikul *et al.* 1987.

RESULTS AND DISCUSSION

Two compounds were isolated from the leaves of *Orophea polycarpa*, one of them being converted to the other on standing at room temperature. The former was isolated on column followed by preparative thin-layer chromatography and appeared as faint yellowish crystals, m.p. 95-97°C, which turned to bright yellow on standing even in nitrogenous atmosphere. The mass spectrum having M⁺ at m/z 340 was consistent with molecular formula of C₁₈H₁₈O₅. Further scrutiny of the UV, IR, ¹H NMR and ¹³C NMR spectra and their comparison with literature confirmed the structure as 2'-hydroxy-4,4',6'-trimethoxychalcone I.

Compound II was isolated as a bright yellow solid, m.p. 115-117°C, having identical molecular weight as the former. The UV, IR, ¹H NMR and ¹³C NMR data were consistent with the reported data for 5,7,4'-trimethoxyflavanone. It is worthwhile noting that interconversion between 2'-hydroxychalcones and the corresponding flavanones in the presence of acid is a known phenomenon (Sheshadri 1962). Our observation of the conversion of 2'-hydroxy-4,4',6'-trimethoxychalcone to 5,7,4'-trimethoxyflavanone on standing may imply the possibility of the former as an artifact. This possibility was however not verified in this study.

Naturally occurring 5,7,4'-trimethoxyflavanone or naringenin trimethyl ether has been isolated from *Dahlia tenuicaulis* (syn. *D. lehmanni*) (Kaufmann and Lam 1967; Lam and Wrang 1975). Isolation of 2'-hydroxy-4,4',6'-trimethoxychalcone was, however, first reported from *Piper methysticum* (Hansel *et al.* 1963; Dutta and Som 1978). Subsequently it has also been reported to occur in *D. tenuicaulis* (Lam and Wrang 1975), *Boesenbergia pandurata* (Herunsalee *et al.* 1987), *Boesenbergia* sp. nov. (Tuntiwachwuttikul *et al.* 1987) and *Kaempferia angustifolia* (Pancharoen *et al.* 1989). Our literature search indicates that these flavonoids have not previously been isolated from the genus *Orophea*.



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