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한라봉 과피로부터 기능성 식물성분의 분리 동정

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Phytochemical Constituents from the Peels of Hallabong

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Objectives

Isolation of phytochemical constituents from the peels of Hallabong ([*Citrus unshiu* Marc. × *C. Sinensis* Osbeck] × *C. reticulata* Blanco) and structure elucidation of them.

Materials and Methods

○ Plant materials : The peels of Hallabong

○ Methods :

The air-dried peel (3096.7 g) of Hallabong was extracted seven times with methanol (8000 ㎖ × 7) under reflux. After filteration using filter paper (No. 2, 600 × 600 mm), the filterate was concentrated *in vacuo* to produce an methanol extract (1650.2 g). The extracts (727.4 g) thus obtained was suspended in distilled water and partitioned in turn using *n*-hexane, chloroform, ethyl acetate, and *n*-butanol.

A portion of the *n*-hexane (7.8 g) fraction was chromatographed on a silica gel column (No. 7734, 6 × 80 cm) using stepwise gradient *n*-hexane and ethyl acetate solvent system (100 : 0 → 0 : 100) to afford sub-fractions. Among them, a gradient of 75 : 25 yielded compound **1**. The sub-fraction 17 was dissolved in chloroform (3.0 ㎖) and subjected to a recycling preparative HPLC. Through recycling and separating the peak, this procedure yielded compound **2**. A part of the chloroform fraction (5.6 g) performed a silica gel open column chromatography using stepwise gradient *n*-hexane and ethyl acetate solvent system (100 : 0 → 0 : 100). Compound **3** was yielded at a solvent gradient of 35 : 65.

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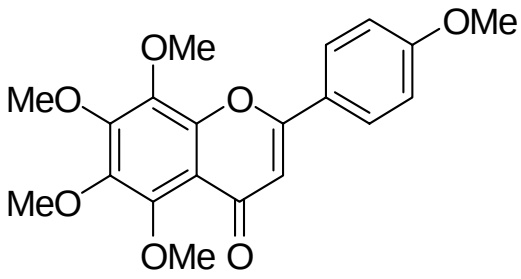
Results

○ Compound **1** was obtained as pale yellow powder. In the EI-MS of **1**, the molecular ion peak showed at *m/z* 372 corresponding to the molecular formula $C_{20}H_{20}O_7$. In the ^{1}H-NMR spectrum of **1**, the singlets of five -OMe signals at δ 3.78, 3.84, 3.85, 3.96 and 4.02 and aromatic proton signals at δ 6.75 were observed. Two doublets of symmetric structure in aromatic ring signal at δ 7.14, 7.99 (J = 9.0 Hz) were observed. Consequently, compound **1** was identified as a flavonoid, tangeretin.

○ Compound **2** was obtained as white amorphous form. In the EI-MS of **2**, the molecular ion peak showed at *m/z* 414 corresponding to the molecular formula $C_{29}H_{50}O$. In the ^{1}H-NMR spectrum of **2**, the singlet of angular methyl signals at δ 0.68, 1.01 were observed. The characteristic peaks of β-sitosterol were observed at δ 3.53 (m), 5.36 (br.s, J = 5.1 Hz). From these data, compound **2** was identified as a sterol, β-sitosterol.

○ Compound **3** was obtained as white amorphous form. Compound **3** differs from **2** because the pattern of fragment peaks and the Rf values using TLC analysis are not same. Through analysis of FAB-MS, the quasimolecular ion peak showed at *m/z* 577 ([M^++H]). In the ^{1}H-NMR spectrum of **3**, the signals were similar to ^{1}H-NMR peaks of compound **2**. But, The signals from δ 3.94 to 4.61 were different because of glucose. From a comparison of these data with literature, compound **3** was identified as a sterol glucoside, daucosterol.



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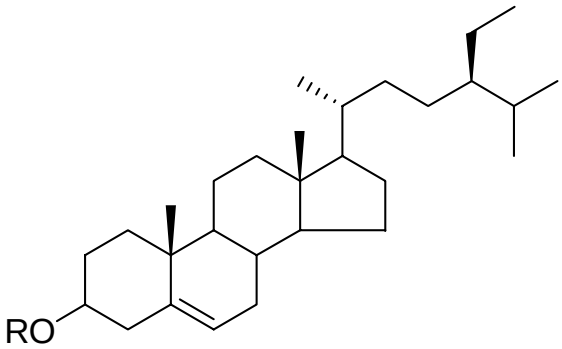

2 R = H
3 R = Glc

Fig. 1. Chemical structures of a flavonoid (**1**) and two sterols (**2** and **3**) isolated from the peels of Hallabong