Ⅱ-11

Phytochemical Constituents from the Fruits of Tartary Buckwheat

Department of Integrative Plant Science, Chung-Ang University, Anseong 456-756, Korea : Jeong Min Lee, Hye Min Kim, Dong Gu Lee, So-Youn Mok, Sanghyun Lee*
Highland Agriculture Research Center, National Institute of Crop Science, Pyeongchang 232-955, Korea : Young-Ho Yoon

쓴메밀 종자로부터 성분 분리 및 구조 동정

중앙대학교 : 이정민, 김혜민, 이동구, 목소연, 이상현*
국립식량과학원 : 윤영호

Objective

Isolation and identification of phytochemical constituents from the fruits of tartary buckwheat.

Materials and Methods

○ Plant materials : The fruits of tartary buckwheat
○ Methods :
The air-dried fruits of tartary buckwheat (*Fagopyrum tataricum*) (9995.2 g) were grounded into powder and extracted with MeOH (4 L × 5) under reflux. The MeOH extracts (399.8 g) was recrystallized with MeOH to afford compounds **1** and then the residue was suspended in H_2O and partitioned with *n*-hexane (54.2 g) and CH_2Cl_2 (15.9 g).
A portion of the *n*-hexane fraction (54.2 g) was chromatographed on a silica gel (6 × 80 cm, No. 7734) column eluting with a gradient of *n*-hexane-EtOAc = 90:10, 100% EtOAc to afford compounds **2** and **3**, respectively.
A portion of the CH_2Cl_2 fraction (15.9 g) was chromatographed on a silica gel (6 × 80 cm, No. 7734) column eluting with a gradient of *n*-hexane-EtOAc = 70:30 to afford compounds **4**.

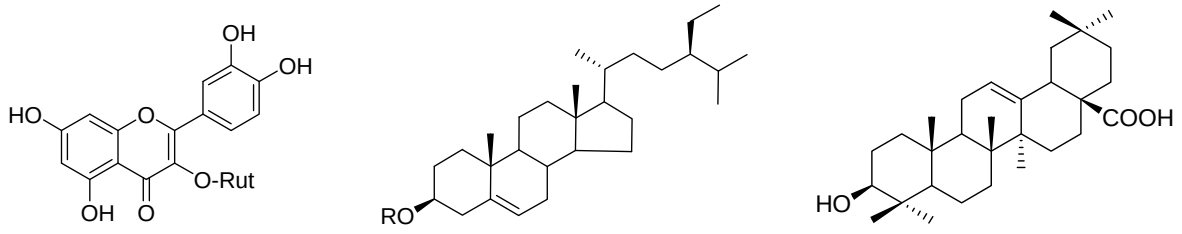
* Corresponding author: Sannhyun Lee Tel: 031-670-4688 e-mail: slee@cau.ac.kr

Results

○ Compound **1** was obtained as yellow powders from the MeOH extracts and it showed a molecular ion peak at *m/z* 611 $[M+H]^+$ in the FAB-MS. The typical 5-OH flavonoid signals of **1** were observed in the ^{1}H-and ^{13}C-NMR spectrum. In the ^{1}H-NMR spectra, it had an ABX system (H-2′, -5′ and -6′), as demonstrated by the coupling constant signal at δ 7.54 (d, H-2′), 7.55 (dd, H-6′), and 6.84 (d, H-5′) in the B-ring structure. In the ^{13}C-NMR spectra, the carbonyl carbon sgnals of the C-ring were observed at δ 177.3 and anomeric carbon signals were observed at δ 17.7-98.7. In the ^{1}H-NMR spectra, due to the anomeric proton of glucose and rhamnose shown δ 5.34 and 5.27, respectively. The rutinoside position was at C-3 of aglycone. Accordingly, the structure of **1** was elucidated as rutin.

○ Compounds **2** and **3** were obtained as white powders from *n*-hexane fraction. In the ^{1}H-NMR spectra of **2** and **3** showed existence of sterol skeleton. The olefinic proton broad doublet one signal at δ 5.34-5.35 was showed H-6. In the ^{13}C-NMR specta of **2** and **3** showed 27-33 resonances, C-5 and C-6 signals were observed at δ 141.0-141.3 and 121.9-122.3, respectively. Compounds **2** and **3** had similar structural signals. However, the difference between **2** and **3** were in the presence of the glucose. The anomeric proton of glucose showed at δ 4.11 (d, *J*=7.8Hz), glucose position was at C-3 (β-linkage) of aglycone. Accordingly, the structure of **2** and **3** were elucidated as β-sitosterol and daucosterol.

○ Compound **4** was obtained as white powders from the CH_2Cl_2 fraction. In the EI-MS spectrum of **4**, molecular peak showed at *m/z* 456 $[M]^+$ corresponding to the molecular formula $C_{30}H_{48}O_3$. In the ^{1}H-NMR spectra of **4** , seven tertiary methyl group signals at δ 0.74-1.30 (s, H23-27, 29-30), one olefinic proton signals at δ 5.26 (H-12), one oxygen bearing methane proton signals at δ 3.23 (dd, H-3). The typical oleane-12-ene-skeleton terpenoids signals of **4** were observed in the ^{1}H-NMR spectrum. Accordingly, the structure of **4** was elucidated as oleanolic acid.



1 **2** (R=H) and **3** (R=Glc) **4**

Fig. 1. Structures of compounds **1-4**