**Reviewer’s Comments**



 **PHYTOCHEMISTRY STUDY OF PLANTS BELONGING TO *CAPPARIS***

ABSTRACT

The research work included in the present paper is mainly focused on isolation and structure elucidation of pure chemical constituents from the selected plants belong to *Capparis* genera. describes the phytochemical investigation of *C.* cartilaginea. Three new compounds as (*Kaempferol*, 3,23-Dihydroxy-lup-20(29)-en-28-oic acid & *β*-Sitosterol ) were isolated from the plant. Various spectroscopic techniques were used to characterize their structures.

Keywords— *Capparis* cartilaginealeaves; *Kaempferol*, 3,23-Dihydroxy-lup-20(29)-en-28-oic acid & *β*-SitosterolNMR analysis

**Introduction**

Products of natural origins can be called “natural products” which include an entire organism such as plants, animals, or microorganisms that has not been subjected to any kind of processing or treatment other than a simple process of preservation (e.g. drying), an extract of an organism, exudates and isolated secondary metabolites such as alkaloids, coumarins, flavonoids, glycosides, lignans, steroids, sugars, terpenoids, etc. from the plants, animals, or microorganisms (1). Concepts of secondary metabolism include products of overflow metabolism as a result of nutrient limitation, shunt metabolism produced during idiophase, defense mechanism regulator molecules, etc. (2). Medicinal plants used as treatment of diseases and therapeutic agents for the management of health because they have power over health promoting effects and have bioactive chemical components (3). Capers is a medicinal plant and native in the dry areas of Western or Central Asia, found growing wild all over Mediterranean ( France, Spain, Italy and Algeria); furthermore, the plant is found in Iran, Iraq, Cyprus, Greece, Yemen and Egypt (4,5). Capers has medicinal distinctive such as rheumatism. Roots used as diuretic, astringent, and tonic (6). Bark root used as appetizer, astringent, tonic, antidiarrheic and treat hemorrhoids and spleen disease. Bark was also used for gout and rheumatism, as expectorant, and for chest diseases. Infusion of stems and root bark were used as antidiarrheic and febrifuge. Fresh fruits were used in sciatica, and dropsy (4,7).

MATERIALS AND METHODS

*Plant material*

collection the plant 26-11-2017 from Al-Mahweet North City in Yemen. Identification and classification of the plant material was performed at the Faculty of Medical Science of the University of Al-Razi specimens were pressed and A voucher specimen (CCJ017) was deposited in a collection housed at the Department of Pharmacy and Pharmacology.

Extraction and Isolation.

The air dried aerial parts of *CAPPARIS CARTILAGINEA* (7 kg) were extracted at room temperature with methanol (x3; 4 days). The combined methanolic extract was evaporated under vacuum pressure to yield g of the residue, which was then partitioned with organic solvent (DCM, EtOAc and MeOH) by addition of H2O to obtained dichloromethane (9 g), ethyl acetate (26 g), and aqueous methanolic (30 g) fractions.

The EtOAc fraction (26 g) was chromatographed on silica gel (70-230 mesh, 1500 g, 9 x 120 cm) column, eluting with ethyl acetate and methanol, in order to increase the polarity of solvent to methanol 100% as eluent and each collected fraction was 20 mL. Fractions 4 to 11 were combined and rechromatographed by radial chromatography to yielded CCI (6.00 mg) identified as *Kaempferol* (1), CCII (3.8 mg) identified as 3,23-Dihydroxy-lup-20(29)-en-28-oic acid (2) & CCIII (5 mg) identified as *β*-Sitosterol (3), were identified by comparison with data from previous NMR and MS spectra.

*Kaempferol* (1):- yellowish powder. 1H NMR (CD3COCD3, 500 MHz): δ 8.05 (2H, d, H-2ʹ,6ʹ), 6.89 (2H, d, H-3ʹ,5ʹ), 6.21 (1H, d, H-6), 6.41 (1H, d, H-8), 5.27 (1H, d, H-1' ). 13C NMR (CD3COCD3, 150 MHz): δ 179.8 (C-4), 166.0 (C-7), 163.1 (C-5), 160.8 (C-4' ), 158.5 (C-2), 156.9 (C-9), 136.0 (C-3), 133.5 (C-2' ),131.9 (6' ), 123.0 (C-1' ), 115.9 (C-3', 5' ), 106.1 (C-10), 100.9 (C-6), 93.1 (C-8).



Fig. (1), *Kaempferol*

3,23-Dihydroxy-lup-20(29)-en-28-oic acid (2):- white amorphous powder. 1H NMR (CD3COCD3, 500 MHz): δ 4.62, 4,74 (2H, d, H-29a, b), 4.86 (1H, m, H-3), 3.10, 3.45 (2H, d, H-23), 0.79, 0.96, 0.98, 1.03, 1,66 ( each 3H, s, Me×5). 13C NMR (CD3COCD3, 150 MHz): δ 179.6 (C-28), 150.3 (C-20), 111.1 (C-29), 76.1 (C-3), 68.5 (C-23), 53.5 (C-5), 51.6 (C-9), 49.5 (C-18), 48.0 (C-19), 44.2 (C-17), 43.1 (C-14), 40.9 (C-8), 40.1 (C-22), 39.0 (C-13), 38.4 (C-4), 37.4 (C-1), 37.0 (C-10), 36.5 (C-16), 34.5 (C-7), 31.0 (C-21), 28.2 (C-23), 27.3 (C-15), 26.9 (C-12), 25.3 (C-2), 22.1 (C-11), 20.5 (C-30), 18.5 (C-6), 18.2 (C-28), 16.3 (C-25), 16.0 (C-26), 15.4 (C-24), 14.8 (C-27).



Fig. (2), 3,23-Dihydroxy-lup-20(29)-en-28-oic acid

 *β*-Sitosterol (3):- white amorphous powder, 1H NMR (CD3COCD3, 500 MHz): δ 0.68, 0.77, 0.80, 0.89, 0.96 and 1.07 (each 3H, s, Me × 6), 3.93 (1H, m, H-3), 5.41 (1H, t, H-6), 13C NMR (CD3COCD3, 150 MHz): δ 141.4 (C-5), 120.6 (C-6), 77.8 (C-3), 57.0 (C-14), 56.1 (C-17), 51.4 (C-24), 50.3 (C-9), 46.0 (C-25), 42.4 (C-13), 40.7 (C-20), 39.8 (C-12), 37.5(C-4), 37.4 (C-1), 36.7 (C-10), 34.1 (C-22), 32.1 (C-8), 31.9 (C-7), 30.5 (C-23), 29.2 (C-16), 28.4 (C-2), 25.6 (C-28), 24.5 (C-15), 21.4 (C-21), 21.3 (C-11), 20.0 (C-27), 19.6 (C-26), 19.1 (C-19), 12.2 (C-29), 12.1 (C-18).



*Fig (3), β*-Sitosterol

*Result and Discussion*

Compound (1);

was isolated as yellowish powder. The IR spectrum showed the presence of hydroxyl (3395 cm-1) and carbonyl (1707 cm-1) groups. Its molecular formula (C16H20O5). 1H-NMR spectrum the chemical shifts and the coupling constants of protons indicated a 5,7-dihydroxylated pattern for ring A (two *meta*-coupled doublet at *δ*H 6.21 (1H, d, H-6) and 6.41 (1H, d, H-8)) and a 4ʹ- hydroxylation for ring B (two *ortho*-coupled doublet signals at *δ*H 8.05 (2H, d, H-2ʹ,6ʹ) and 6.89 (2H, d, H-3ʹ,5ʹ). 13C NMR showed fifteen carbon signal including six methins and nine quaternary carbons. The alkenes carbons appeared at δ160.0 and 135.2.

Compound (2); was isolated as white amorphous powder. Its molecular formula (C30H50O4) was established by 1H- and 13C-NMR analysis. The 1H-NMR spectrum exhibited the presence of five tertiary methyl groups at *δ* H 0.79 (3H, s), 0.96 (3H, s), 0.98 (3H, s), 1.03 (3H, s) and 1.66 (3H, s) at C-24, C-25, C-26, C-27, and C-30 positions respectively.

The 13C-NMR spectrum of compound showed an ester carbonyl, five tertiary methyl groups, a carboxylic acid carbon and a terminal double bond. In addition to these signals, twenty three other carbon resonances were found in 13C-NMR spectrum.

Compound (3); was purified as white amorphous powder. 1H NMR spectra showed the presence of six methyl’s appeared at δ 0.68, 0.77, 0.80, 0.89, 0.96 and 1.07. The proton of H-3 appeared as a multiplet at δ 3.93. It also showed olefinic protons at δ 5.41. 13C NMR showed twenty nine carbon signal including six methyles, nine methylenes, eleven methins and three quaternary carbons. The alkenes carbons appeared at δ141.40 and 120.6.

**Conclusion**

the known compound **;** *Kaempferol*, 3,23-Dihydroxy-lup-20(29)-en-28-oic acid & *β*-Sitosterolfrom the leaves of *Capparis* cartilagineaisolation, identification and reported for the first time from this part of the plant. The work was carried out by means of various physical (solvent extraction, column chromatography, radial chromatography, preparative TLC and malting points) and spectral techniques.

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