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Sesquiterpenes Compounds from the Leaves of *Capparis cartilaginea*

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Abstract:
An examination of methanol extract of *Capparis cartilaginea* leaves showed isolation of 4 terpenoid compounds characterized as 3,9-Dihydroxygermacra-4,10,11-triene, 6-Methoxyeudesm-4-en-1-ol, Gallic acid, and 3,4-Dihydroxybenzoic acid. Structure elucidations were carried out by the spectroscopy method.

Keywords: *Capparis cartilaginea* leaves, Sesquiterpenes compounds, NMR analysis.

INTRODUCTION

The medicinal plants have been used as healing agents and treatment of diseases since prehistoric times (Locatelli *et al.,* 2014). Now the plants are used as traditional medicine in developing and industrialized countries. The herbal remedy is one, in which the main therapeutic activity rests on plant metabolites (Shahzad *et al.,* 2017; Ullah *et al.,* 2018). Traditional medicine refers to health practices, approaches, knowledge, and beliefs incorporating plant, animal, and mineral-based medicines and comprises medical knowledge systems that developed over generations within various societies before the era of modern medicine (Gunjan *et al.,* 2015). Medicinal plant parts are used for the extraction of raw drugs as they have diverse medicinal properties (Al-Mahweety, 2016a; Al- Mahweety, 2016b; Ali *et al.,* 2017; Hussain *et al.,* 2016; Iqbal and Ashraf, 2019; Kalim *et al.,* 2016; Shuaib *et al.,* 2019). The phytochemical studies of capers extract demonstrated the occurrence of many chemical compounds with very exciting genetic activities (Tagnaout *et al.,* 2016) which include alkaloids, fatty acids, phenolic acids, flavonoids, aldehydes, esters, vitamins, and glucosinolates (Matthaus and Ozcan, 2002). *Capparis spinosa* aqueous extracts show antifungal characteristics against *Trichophyton violaceum* (Gadgoli and Mishra, 1999). Besides, the study of the different parts of *C. spinosa* L., showed biological activities as well as anti-inflammatory, anti-diabetic, antihistamine effects, and anti-allergic effects (Eddouks *et al.,* 2004; Trombetta *et al.,* 2005; Ullah *et al.,* 2018).

Capper contains sulfides, isothiocyanates, and cyclooctasulfur. *Capparis* are shrubs, trees, and woody climbers comprising about 250-400 species (Inocencio *et al.,* 2006), found in tropical and subtropical zones of Southern America, Europe, Africa, Madagascar, Asia, Australia, and the Pacific Islands (WHO, 2013; Locatelli *et al.,* 2017). *Capparis cartilaginea* Decne, *C. spinose*, *C. deciduas,* and *C. tomentosa* were found in Yemen (Al-Khulaidi, 2013). *Capparis cartilaginea* has various traditional uses in the Arab region. It is used for easing bruises, childbirth, earache, headache, paralysis, swelling, skin, and joint inflammation, knee problems tendinitis, and snakebites (Locatelli *et al.,* 2017). It is called lattssaf, laşaf, or nişaf (Al-Duais, 2007) and used to treat itching, shortness of breath, head cold, tumors, wounds, boil, and for painful knees (Locatelli *et al.,* 2017). The current study aimed to investigate the phytochemical screening of leaves of *Capparis cartilaginea*.

MATERIALS AND METHODS

Collection of Plant Material

The plant leaves were collected from AlMahweet (Yemen). Identification of the plant was performed at the Faculty of Medical Science, University of Al-Razi. The specimens were preserved and the sample (CCJ017) was deposited in a collection housed at the Department of Pharmacy and Pharmacology.

Extraction and Fractionation

The leaves (7000 g), of the plant, were cleaned, air-dried in the shade, and then grounded. The parts were extracted with methanol and filtered. The filtrate was partitioned with organic solvent (DCM, EtOAc, and MeOH) by the addition of H_2O yielding dichloromethane (9 g), ethyl acetate (26 g), and aqueous methanolic (30 g) fractions.

Aqueous methanolic (30 g) was fractioned and chromatographed by column chromatography to yield CCJI (5.5 mg) identified as (3,9-Dihydroxygermacra-4,10,11- triene, CCJII (4.4 mg) identified as 6- methoxyeudesm-4 -en-1-ol, CCJIII (6 mg) identified as Gallic acid and CCIV (6 mg) identified as 3,4-Dihydroxybenzoic acid, recognized by comparison with data from earlier NMR and MS spectra.

(1): 3,9-Dihydroxygermacra-4,10,11- tri-ene.

Colorless stick of gum; ^{1}H-NMR ($CDCL_3$) δ 1.62 (2H, m, H-6), 1.71 (3H, s, H-13),1.80 (2H, m, H-8), 2.10 (2H, m, H-7), 2.21 (2H, m, H-1), 2.31 (2H, m, H-2), 2.38 (2H, m, H-5),4.03 (1H, m, H-9), 4.23 (1H,m, H-3), 4.86 (2H, s, H-12), 5.08 (2H, s, H-14), 5.14 (2H, s, H-15.^{13}C NMR ($CDCl_3$):δ 151.09 (C-10), 149.65 (C-11), 148.68 (C-4), 115.06 (C-14), 113.97 (C-15), 109.94 (C-12), 77.20 (C-9), 73.96 (C-3), 46.06 (C-7), 40.09 (C-8), 33.24 (C-6), 32.63 (C-2), 31.05(C-5), 24.30 (C-1), 20.09 (C-13).

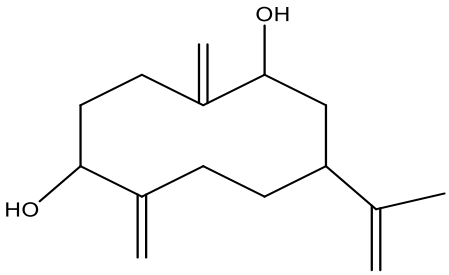

Fig.1. Structure of 3,9-Dihydroxygermacra-4,10,11-triene

(2): 6- Methoxyeudesm- 4 -en-1-ol.

White powder; ^{1}H-NMR ($CDCL_3$) δ 1.01 (3H, s, H-12, 13), 1.11 (3H, s, H-14), 1.62 (1H, m, H-6), 1.80 (2H, m, H-8),1.93 (1H, m, H-9), 2.10 (1H, m, H-7), 2.23 (1H,m, H-3), 2.31 (2H, m, H-2), 2.38 (1H, m, H-5), 3.14 (1H, m, H-1), 3.29 (3H, s, OCH_3), 5.14 (2H, s, H-15). ^{13}C NMR ($CDCl_3$): δ 145.35 (C-4), 110.15 (C-10), 82,05 (C-6), 79.10 (C-1), 56.95(C-OCH_3), 54.24 (C-5), 46.50 (C-7), 43.06 (C-10), 36.08 (C-3), 33.94 (C-9), 32.30 (C-2), 25.45 (C-13), 20.76 (C-12, 13), 19,92 C-8), 14.04 C-14).

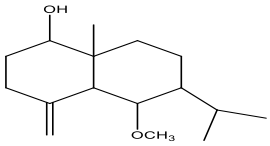

Fig. 2. Structure of 6- methoxyeudesm- 4 -en-1-ol

(3): Gallic acid.

White crystals, ^{1}H-NMR ($CDCL_3$); δ 7.37 (2H, s, H- 2, 6). ^{13}C NMR ($CDCl_3$): δ 172.08 (C-7), 148.07 (C-3, 5), 139.83 (C-4), 123.45 (C-1), 110.90 (C-2, 6).

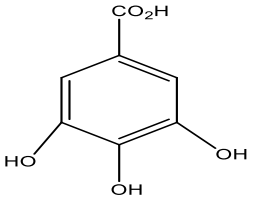

Fig.3. Structure of Gallic acid 3

(4): 3, 4-Dihydroxybenzoic acid.

White powder. ^{1}H-NMR ($CDCL_3$); δ 6.82 (1H, d, H- 2), 7.52 (1H, m, H-2), 7.95 (1H, m, H-6). ^{13}C NMR ($CDCl_3$): δ 171.82 (C-7), 149.85 (C-4), 143.83 (C-3), 123.67 (C-1, 6), 118.64 (C- 2), 114.72 (C-5).

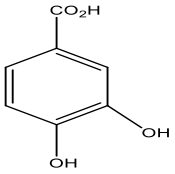

Fig. 4. Structure of 3, 4-Dihydroxybenzoic acid

RESULTS AND DISCUSSION

The leaves extract of *C. cartilaginea* is a potential source of bioactive compounds that could have a role in anti-inflammation. The compounds obtained from plants can be a therapeutic alternative for microbial diseases in an era of progressive antibiotic resistance (Ali *et al.,* 2017; Iqbal and Ashraf, 2019). Compound [1] appeared as a colorless stick of gum,^{1}H-NMR spectrum of the compound displayed signals for two oxygenated methine proton signals at 4.03 (1H, m, H-9) and 4.23 (1H,m, H-3), two exomethylenes at δ 5.08 (2H, s, H-14), 5.14 (2H, s, H-15), one isopropenyl proton δ 1.70 (3H, s, H-13) and 4.86 (2H, s, H-12). The ^{13}C-NMR spectrum showed fifteen carbon signals including

olefinic carbons at δ 151.09 (C-10), 149.65 (C-11), 148.68 (C-4), 115.06 (C-14), 113.97 (C-15) and109.94 (C-12). One methyl carbon at δ20.09 (C-13), two oxygenated carbons at δ77.20 (C-9), and 73.96 (C-3).

Compound [2] was white powder. The NMR spectral data of the compound have signals for one oxygenated methine proton signals at 3.14 (1H, m, H-1), one exomethylenes at δ 5.14 (2H, s, H-15). ^{13}C-NMR data appeared sixteen carbon signals including olefin carbons at δ 145.35 (C-4), 110.15 (C-10). One methyl carbon at δ20.09 (C-13), two oxygenated carbons at δ 79.08 (C-1).

Compound [3] appeared as white crystals, Molecular analysis by ^{1}H- and ^{13}C-NMR determined its molecular formula ($C_7H_6O_5$). The ^{1}H-NMR spectrum of the compound showed the benzene ring signal at *δ* 7.13 (2H, H- 2, 6). The ^{13}C-NMR spectrum contains carbon of carbonyl at *δ* 170.2 (C-7), carbons (1, 3, 5, and 4) quaternary at 122.6, 147.2, 140.5, and two carbons (2, 6) at 111.5.

Compound [4] was isolated as white powder, displayed ^{1}H-NMR spectrum for aromatic ring signal at *δ* 7.8 (H, m, H- 6), 7.5 (2H, m, H-2), 6.7 (H, d, H-5). The ^{13}C-NMR spectrum showed carbonyl carbon at *δ* 169.5 (C-7), three quaternary carbons at 121.7 (C-1), 144.8 (C-3), 150.5 (C-4), and three methine carbons at 113.4 (C-5), 116.8 (C-2), 125.3 (C-6). Several studies have reported the isolation of medicinal compounds from various plants (Al- Mahweety, J.A.N., 2016a,b; Trombetta *et al.,* 2005).

CONCLUSION

3,9-Dihydroxygermacra-4,10,11- triene, 6-Methoxyeudesm- 4 -en-1-ol, Gallic acid, and 3,4-Dihydroxybenzoic acid were isolated and identified from the leaves of *Capparis cartilaginea* by using different physical (solvent extraction, C. C., TLC) and spectral techniques.

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CONFLICT OF INTEREST

There is no conflict of interest.

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