INTRODUCTION

Medically significant genus Caralluna is widely studied for its stem and fruits. It is belong to the family Asclepiadaceae, which comprises 200 genera and 2500 species. About 200 species belong to genus Caralluma distributed throughout Africa and Asia. The greater part of species are native in Indian sub-continent and Arabian Peninsula. A number of Caralluma species use as anti-hyperglycemic goings-on of their crude extracts or their corresponding fractions. The investigation of the chemical and biological members of genus Caralluma the anti-hyperglycemic activity of the extracts, fractions of the major pregnane glycoside of the aerial parts of C. quadrangula was investigated in Kingdom Saudi Arabia as novel. We use the extract of C. quadrangula as herbal medicine in Saudi, for the treatment of freckles, diabetes, vitiligo and melasma and for thirst, hunger. Several countries the species of Caralluma are fit to be eaten and variety division for the traditional medicine organisation. These plants can be use as folk medicine as remedies to health situation and treat large multiplicity of diseases. The species of C. arabica use as traditionally for an emollient and diuretic In United Arab of Emirates. Also used to care for diabetes, hypertension and liver diseases. The C. Arabica flower used for wounds and cuts, while the juice of the stem is given to sick people to speed convalescence of burns, itchy skin and sunburns. The C. attenuate species in Indian (Andhra Pradesh) use for eaten raw as an anti-diabetic agent, although the juice of the plant beside the black pepper is suggested in the treatment of migraine. The different applications of Caralluma plants in folk medicine have prompted the phytochemical and biological investigations of their constituents. The pregnant glycosides, flavone glycosides, megastigm-anene glycosides, bitter principles, triterpenes and saponins isolated from Caralluma.

MATERIALS AND METHODS

Purified every one of chemical constituent by subsequent standard procedures and all chemicals used systematic Reagent evaluation.

Plant material:-

Roots of Caralluma quadrangula (Asclepiadaceae) were collected from Sana'a 2014. The plant identified
by Dr. Hessen Ibrahim and was deposited voucher sampling of plant in Herbarium, Department of Phytochemistry (Sana'a University).

**Extraction and Isolation:**
Shade dried roots were crushed and sieved. Next powder was stored in air closing container. Than weighed and extracted with soxhlet extractor by using solvents Chloroform with consecutive solvent extraction. To concentrate the extracts and removal of final traces of solvent than vapor. After that, recrystallization was done to purify the crude extracts. Melting point was taken by using Fisher-John apparatus. The $^1$H NMR and $^{13}$C NMR spectra were taken on Bruker 100 MHz and 400 MHz, spectrometer, using an internal standard like TMS.

**Extraction and isolation**
Extracted by using Soxhlet (2 Kg) of *C. quadrangula* roots powder with solvents (3X, 8 hours each) and then evaporated collective extracts to give a brown gum residue (8 g) after than separation and purified by silica gel flash column chromatography (FCC) with CHCl$_3$ containing increasing percentages of MeOH as eluent and collected 20 ml for each fraction. Fractions 3-10 were combined and rechromatographed by C.C. with CHCl$_3$-MeOH (8:2) to afford JA1 (4.5 mg) identified as 10a- hydroxyoplopan-4-one (1). CHCl$_3$-MeOH (7:3) to afford JA3 (5.0 mg) identified as 1β, 6α-dihydroxyeudesm-4(15)-ene (2). CHCl$_3$-MeOH (6:4) to afford JA4 (5.0 mg) identified as and CHCl$_3$-MeOH (3:7) to afford JA4 (7.0 mg) identified as quercetin- rhamnopyranosyl- D-glucopyranose (Rutin) (4). NMR data used to identified for each pure compounds.

**Figure 1:** 10α-Hydroxyoplopan-4-one (1)

$^1$H-NMR (100 MHz, CDCl$_3$): δ 2.75 (1H, m, H-3), 2.30 (3H, s, H-15), 1.50 (3H, s, H-13), 1.10 (3H, d, J= 7), 0.87 (3H, d, J= 12); $^{13}$C-NMR (MHz, CDCl$_3$): δ: 209.4 (C-14), 73.0 (C-8), 56.0 (C-3), 54.6 (C-9), 48.2 (C-5), 45.8 (C-4), 41.0 (C-7), 28.6 (C-10), 27.5 (C-1), 24.4 (C-2), 21.9 (C-6), 21.0 (C-11), 19.2 (C-13), 18.5 (C-15), 15.0 (C-12).

**Dihydroxyeudesm-4(15)-ene (2).** $^1$H-NMR (100 MHz, CDCl$_3$): δ: 5.10 (1H, brs, H-15), 5.01 (1H, brs, H-15), 3.79 (1H, t, J= 6β), 3.42 (1H, dd, J= 1Hα), 2.33 (1H, ddd, J= 3α), 2.24 (1H, sept, J= 11), 2.07 (1H, ddd, J= 3β), 1.91 (1H, s, H-8), 1.85 (1H, ddd, J= 2α), 1.75 (1H, brd, J= 5α), 1.53 (1H, m, H-2β), 1.53 (1H, m, J= 8), 1.43 (1H, brs, 1-OH), 1.28 (1H, m, H-7α), 1.20 (1H, m, J= 9a), 1.18 (1H, m, J= 9b), 1.02 (3H, d, J= 13), 0.87 (3H, d, J= 12), 0.72 (3H, s, H-14); $^{13}$C-NMR (MHz, CDCl$_3$): δ: 147.4 (C-4), 108.2 (C-15), 79.1 (C-1), 67.8 (C-6), 56.4 (C-5), 50.1 (C-7), 42.1 (C-10), 36.9 (C-9), 36.1 (C-3), 32.2 (C-2), 26.5 (C-11), 21.8 (C-13), 19.1 (C-8), 16.6 (C-12), 12.0 (C-14).

**Figure 2:** dihydroxyeudesm-4(15)-ene (2)

**Quercetin-L-rhamnopyranosyl-(1→6)-Dglucopyranose (3).** $^1$H NMR (100 MHz, CDCl$_3$): δ 6.22 (1H, d, J= 6), 6.40 (1H, d, J= 8), 7.68 (1H, s, J= 2), 6.90 (1H, d, J= 5), 5.79 (1H, d, J= 6), 5.10 (1H, d, J= 1Hα), 3.49 (1H, m, J= 2), 3.34 (H, m, J= 3), 3.50 (1H, m, J= 4), 3.30 (1H, m, J= 5), 4.51 (1H, br, J= 1), 1.30 (1H, m, J= 2), 3.43 (1H, m, J= 3), 3.54 (1H, m, J= 4), 3.31 (1H, m, J= 5), 1.17 (3H, d, J= 6), 1.35 (C NMR (CDCl$_3$)): δ 158.1 (C-2), 135.1 (C-3), 180.0 (C-4), 160.0 (C-5), 100.0 (C-6), 166.1 (C-7), 95.1 (C-8), 163.0 (C-9), 104.7 (C-10), 123.0 (C-11), 186.2 (C-12), 146.1 (C-13), 150.1 (C-14), 115.8 (C-15), 123.4 (C-6), 105.0 (C-1), 75.6 (C-2), 77.8 (C-3), 74.9 (C-4), 77.2 (C-5), 69.0 (C-6), 103.1 (C-7), 72.1 (C-8), 74.1 (C-9), 70.4 (C-10), 19.2 (C-11).

**Figure 3:** Quercetin -rhamnopyranosyl-D-glucopyranose

**Figure 4:** H$^1$ NMR of 10α- hydroxyoplopan-4-one (1)
RESULTS AND DISCUSSION

Compound 1: The $^1$H NMR showed one multiplet proton at $\delta$H 2.75 (1H, m), 5.46. High intensity Peaks at $\delta$ 2.30, 1.50, 1.10 and 0.88 are corresponding to methyl groups (Me- (15, 13, 14 and 12), 4 methyl, 4 methylene, 5 methine and 2 quaternary carbons presence in $^{13}$C NMR spectrum. Carboxylic group signals become visible at $\delta$ 209.5. In addition of $\beta$-hydroxyl group to C8 is visible from a peak at $\delta$ 73.1. Hydroxyoplopan-4-one, it has never been isolated before from Caralluma quadrangula, it reported from Cassia buds.

Compound 2: The $^1$H NMR showed signals for three angular methyl singlet's at $\delta$H 0.95, 0.85 and 0.71. Proton of H-6 and H-1 appeared at $\delta$ 3.79 and 3.42. Olefinic protons present at $\delta$ 5.10 and 4.95 for H-15. $^{13}$C NMR showed fifty carbon signal including three CH$_3$, five CH$_2$, five CH and two quaternary carbons. The double bond carbons appeared at $\delta$ 147.4 and 108.1. The significant signal for the 1$\beta$, 6$\alpha$-dihydroxyeudesm-4(15)-ene would be the signals for two carbon attached to hydroxyl group, which is C-1 and C-6 that appeared at $\delta$79.2 and 67.8.
Compound 3: The $^1$H NMR spectrum exhibited signals which were typical of a flavone compound. In addition to the presence of five aromatic protons; one was represented by two meta-coupled protons at $\delta$H 6.23 (d, H-6) and 6.42 (d, H-8). $^{13}$C NMR experiments showed one methyl, 15 methines, one methylene and 10 quaternary carbon atoms, one being the flavone carbonyl (C 180.0)24. NMR spectral data confirmed the sugar part assigned as glucose and rhamnose. A significant downfield shift of the methylene carbon appearing at C 69.1 and assigned to C-6 of glucose, indicated a (1 to 6) type of interglycosidic linkage to the rhamnose moiety. Qercetin-rhamnopyranosyl-D-glucopyranose isolated from C. quadrangula for first time, it was reported in many plants as Taveneria aegyptiaca25,26.

CONCLUSION

The isolation and identification 10α- hydroxyxopan-4-one, dihydroxyudesm-4(15)-ene, and quercetin-rhamnopyranosyl- D-glucopyranose (Rutin), from the roots of C. quadrangula. The work was carried out by means of various physical (solvent extraction, column chromatography, radial chromatography, preparative thin layer chromatography and melting points) and spectral techniques.

AUTHOR’S CONTRIBUTION

The manuscript was carried out, written, and approved in collaboration with all authors.

CONFLICT OF INTEREST

No conflict of interest associated with this work.

REFERENCES