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السلام عليكم ورحمة الله وبركاته .. وبعد ..

نفيدكم بقبول البحث المقدم من قبلكم للنشر بمجلة جامعة الملك عبد العزيز « العلوم » ،

تحت عنوان :

"تحليل GC/MS لمكونات الزيت الطيار لنبات الشيح"

وسنوافيكم بالمسودة لتصحيحها واعادتها الينا بأسرع ما يمكن .

وتقبلوا أطيب تحياتي

رئيس التحرير

أ. د. [signature]

GC/MS ANALYSIS OF THE VOLATILE CONSTITUENTS OF ARTEMESIA MONOSPERMA

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In this article, we describe the volatile constituents of *Artemesia monosperma* Del. from Riyadh area, Saudi Arabia using GC/MS. Twenty five compounds, mainly sesquiterpenes, have been identified. The chemical findings in this article ascertained that *A. monosperma* exhibits ecological variability in its chemical composition.

تحليل المكونات المتطايرة للشيح "أرتميزيا مونوسبيرما" بجهاز GC/MS

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في هـذا البحث تم جمع نبات الشيح "أرتميزيا مونوسبيرما" من منطقة الرياض بالمملكة العربية السعودية وفحصت المكونات الطيارة باستخدام تقنية GC/MS فنتج عن البحث تعريف خمسة وعشرين مركباً معظمها من السسكوي تربينات. وقد جاءت النتائج مؤكدةً أن هذا النبات حساس للتغيرات في موقع الجمع.

Introduction

Many medicinal uses have been reported for the plants of the genus *Artemisia*[1] (Compositae). Saleh reported the insecticidal activity of *A. monosperma* and has attributed this activity to an acetylenic compound[2], which was incorrectly assigned as 3-methyl-3-phenyl-1,4-pentadiyne, but it was later corrected as 2,4-hexadiynylbenzene[3].

A. monosperma was investigated previously for flavonoids[3-6], acetylenes[2,3,7], *p*-hydroxyacetophenone derivatives[3,7-10], coumarins[9], *p*-coumaric acid derivatives[3], tetrahydrofuran-type terpenoids derived from davanone[11], in addition to cycloartenol, β-sitosterol and stigmasterol[11]. Comparing the chemical constituents of aerial parts of two specimens of *A. monosperma*, collected from Saint Katherine, South Sinai, Egypt[11], and from Western Desert, Egypt[3], using the same method of processing, revealed that they are completely different. In this article, we have investigated the chemical constituents of the aerial parts of a third specimen of *A. monosperma*, collected from Riyadh area, Saudi Arabia. The results confirm the variability in the chemical constituents of *A. monosperma* according to locality accessions.

Experimental

GC/MS spectra were taken on QP-7000 Shimadzu, with a fused silica capillary column (30m x 0.25mm ID), film (5% phenyl, 95% methylsilicon) thickness

0.25μ, and the output is an IBM computer with software class 5000 and NIST library for comparison.

The plant material: The aerial parts of *A. monosperma* Del. In the flowering stage (1000g) are collected from Riyadh area in April 1999 and identified by Prof. Dr. A. Faied, Botany Dept., Faculty of Science, King Abdulaziz University. A speciemen was deposited in the Herbarium of Botany Dept., Faculty of Science, King Abdulaziz University.

Processing of plant material: The air-dried, ground aerial parts (1000g) were extracted at room temperature by soaking in a mixture 1:1:1 of methanol/ether/pet. ether 40-60° for 24 hours. The crude extract (84g) was defatted by dissolving in cold MeOH (150 ml) and standing in the fridge freezer for overnight, then, quick filtration and evaporation gave the defatted extract (38g). The defatted extract was fractionated over silica gel (160 g) CC (100 cm length × 4 cm inner diameter) using stepwise elution into six fractions. Am3a (15.8%) and Am3b (10.5%) using the eluent mixture pet. ether/ether 3:1; Am4a (18.4%) and Am4b (14.5%) using pet. ether/ether 1:1; Am5 (11.8%) using ether and Am6 (10.3%) using ether/MeOH 9:1.

Results and Discussion

Fractions described above were found, by ^{1}H-NMR to contain complicated mixtures of weakly polar components. Therefore, GC/MS technique was

subsequently used to analyze the constituents of these fractions. Twenty five compounds were identified based upon the comparison of the mass spectrum given by the computer from the NIST library with the actual spectrum of each component with the standard spectrum from NIST library. After that the structure was further ascertained by comparing their mass spectral data with those available in Adams[12] or of the corresponding compound from the literature.

Fraction Am3a gave β-citronellyl propanoate[12] (21.5%, R_t 29.70 min), geraniol[12] (12.2%, R_t 30.61 min), γ-cadinene[12] (15.8%, R_t 31.64 min), 10(14)-aromadendrene[12] (13.1%, R_t 31.98min), germacrene D[12] (10.2%, R_t 32.25 min) and palmitic acid[13] (9.4%, R_t 38.67 min).

Fraction Am3b afforded β-citronellyl propanoate (18.3%, R_t 29.70 min), spathulenol[12] (4.6%, R_t 30.10 min), caryophyllene oxide[12] (6.6%, R_t 30.22 min), linalool acetate[12] (6.2%, R_t 30.64 min), 1 β-hydroxyallo-aromadendrene[14] (4.8%, R_t 30.94 min), 4(15)-eudesmen-6-ol[14] (3.2%, R_t 31.26 min), methyl ferulate [15] (6.5%, R_t 31.64 min), β-cedrene[12] (7.6%, R_t 32.01 min) and γ-elemene[12] (3.1%, R_t 32.28 min).

Fraction Am4a gave *p*-hydroxyacetophenone[16] (17.9%, R_t 26.17 min), spathulenol (5.5%, R_t 30.10 min), tremetone[3] (5.0%, R_t 33.80 min), 15-hydroxy-α-muurolene[12] (6.5%, R_t 34.83 min), 14-hydroxy-α-muurolene[14] (6.6%, R_t 35.97 min), 4(15)-eudesmen–1,6-diol[14] (10.2%, R_t 37.25 min) and 8,11,13-abietatriene[12] (20.5%, R_t 49.82 min).

Fraction Am4b afforded *p*-hydroxyacetophenone (26.7%, R_t 26.17 min), spathulenol (1.5%, R_t 30.10 min), tremetone (4.9%, R_t 33.80 min), β-patchoulene[12] (3.5%, R_t 34.58 min), trans calamenene[12] (1.4%, R_t 34.80 min), cis calamenene[12] (2.0%, R_t 35.64 min), 14-hydroxy-α-muurolene (10.1%, R_t 35.97 min), 4(15)-eudesmen–1,6-diol (2.1%, R_t 37.25 min), licochalcone B [17] (11.9%, R_t 48.64 min) and 8,11,13-abietatriene (9.9%, R_t 49.82 min).

Fraction Am5a gave 10,11-dihydro-10-hydroxy-tremetone[15] (12%, R_t 42.29 min).

Comparing the chemical findings in the present work with those obtained previously on the same species from other two localities[3,11] revealed that chemical constituents of *A. monosperma* are sensitive to the locality variation. However, the *p*-hydroxyacetophenone derivative tremetone was identified from the specimen of Western Desert, Egypt[3], together with other seven *p*-hydroxyacetophenone derivatives. The specimen of Western Desert seems to be more aromatic and that of Riyadh area is rich in volatile constituents, while that of Saint Katherine[11] is the poorest one in chemical constituents, which were found to be tetrahydrofuran-type terpenoids.

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