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Volatile Constituents of *Trigonella stellate*

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Abstract. In this article, the volatile constituents of *Trigonella stellate* collected from Wadi Qattan, northern east Hazn mountain about 130 km south of Taief, Saudi Arabia, using GC/MS are described. In addition to many fat derivatives, β-sitosterol and stigmasterol, about twenty two volatile compounds were identified. This *Trigonella* species has not been investigated previously for its natural products.

Introduction

Literature survey indicated that *Trigonella stellate* is only screened for flavonoids and saponins[1], but no chemical investigations have been done on the volatile constituents for this species.

From a medicinal point of view, *Trigonella* species were found to be important. Kaur and Kapoor[2] and Langmead *et al.*[3] studied the antioxidant activity of *Trigonella* species. The protective effects of total saponins of *Trigonella foenum-graecum* on acute cerebral ischemia were studied by Li and coworkers[4].

Typical chemical constituents of the genus *Trigonella* are diosgenin[5], furostanol type steroidal saponins[6], flavonoids[7] and flavonol glycosides[8].

Experimental

GC/MS spectra were taken on QP-7000 Shimadzu, with a fused silica capillary column (30 m × 0.25 mm ID), film (5% phenyl, 95% methylsilicon) thickness 0.25 μ, and the output is an IBM computer with software class 5000 and NIST library for comparison.

The Plant Material

The aerial parts of *Trigonella stellate*, in the flowering stage, are collected from Wadi Qattan, northern east Hazn mountain about 130 km south of Taief, in April 2003 and identified by Dr. F. Alghamdi, Botany Dept., Faculty of Science, King Abdulaziz University. A specimen was deposited in the Herbarium of Botany Dept., Faculty of Science, King Abdulaziz University.

Processing of Plant Material

The air-dried, ground aerial parts (300 g) were extracted at room temperature by soaking in a mixture 1:1:1 of methanol/ether/pet. ether 40-60º for 24 hours. The crude extract (6.5 g) was defatted by dissolving in cold MeOH (50 ml) and standing in the fridge freezer for overnight, then, quick filtration and evaporation gave the defatted extract (3.8 g). The defatted extract was fractionated over silica gel (100 g) CC (100 cm length × 4 cm inner diameter) using stepwise elution into five fractions. Fr3a (15.8%) and Fr3b (10.5%) using the eluent mixture pet. ether/ether 3:1; Fr4 (32.4%) using pet. ether/ether 1:1; Fr5a (11.8%) using ether and Fr5b (10.3%) using ether/MeOH 9:1.

Identification of Compounds

Fraction Fr3a afforded by GC/MS lauric acid (R_t 16.20 min, 0.07%), myristic acid (R_t 18.56 min, 0.42%), octadecane (R_t 18.91 min, 0.34%), methyl-pentadecanoate (R_t 19.19 min, 0.08%), pentadecanoic acid (R_t 19.64 min, 0.44%), methyl palmitate (R_t 20.28 min, 3.37%), (*Z*) phytol (R_t 20.50 min, 0.36%), palmitic acid (R_t 21.01 min, 27.57%) methyl heptadecanoate (R_t 21.30 min, 0.47%), heptadecanoic acid (R_t 21.76 min, 1.10%), methyl linoleate (R_t 21.99 min, 1.79%), (*E*) phytol (R_t 22.19 min, 4.49%), methyl stearate (R_t 22.29 min, 0.85%), stearic acid (R_t 22.82 min, 7.28%), nonadecanoic acid (R_t 23.60 min, 1.05%), methyl eicosanoate (R_t 24.14 min, 1.36%), arachidic acid (R_t 24.52 min, 6.52%), methyl heneicosanoate (R_t 25.00 min, 0.73%), heneicosanoic acid (R_t 25.37 min, 0.91%) and docasanoic acid (R_t 26.41 min, 2.80%).

Fraction Fr3b gave by GC/MS 4-hydroxy-3-methoxyacetophenone (R_t 8.00 min, 0.76%), vanillic acid (R_t 9.15 min, 3.12%), dehydronerol **1** (R_t 10.00 min, 0.26%), 4-hydroxy-3,5-dimethoxyacetophenone (R_t 11.16 min, 0.17%), palmitic acid (R_t 13.67 min, 4.89%), (*Z*) phytol (R_t 15.18 min, 1.13%) and stearic acid (R_t 15.65 min, 0.74%).

Fraction Fr4 gave by GC/MS terpin-4-ol (R_t 3.44 min, 1.91%), α-terpineol (R_t 3.62 min, 1.96%), citronellol **2** (R_t 4.15 min, 3.28%), *E*-citral (R_t 4.34 min, 2.30%), geraniol (R_t 4.56 min, 2.17%), dihydromyrcene **3** (R_t 6.06 min, 1.92%),

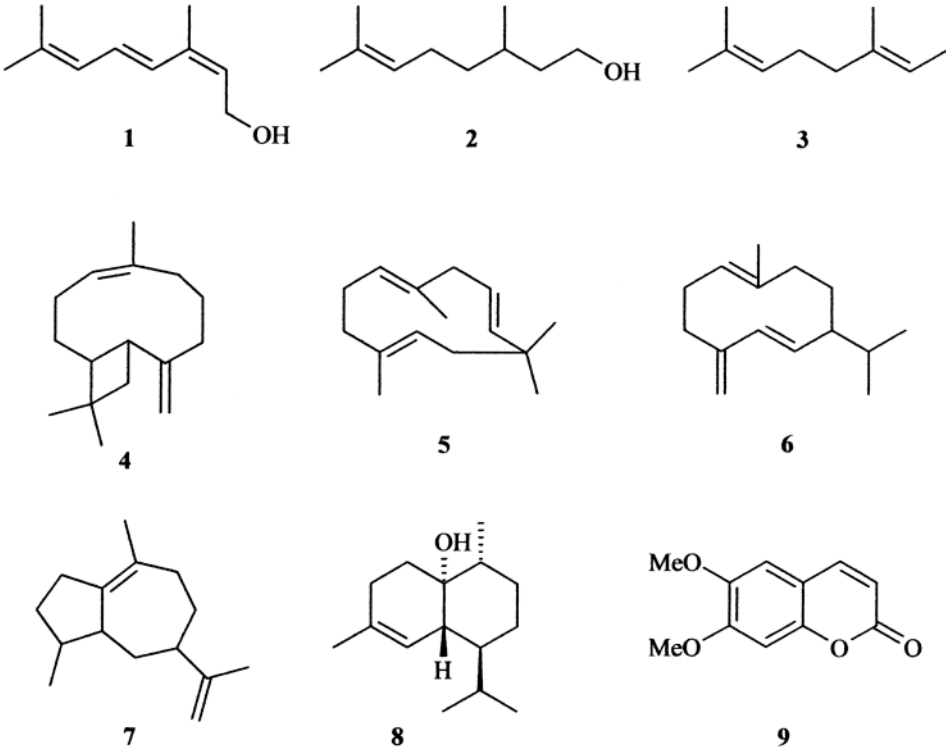
eugenol (R_t 6.14 min, 2.09%), geranyl acetate (R_t 6.52 min, 1.71%), eugenol methyl ether (R_t 6.82 min, 1.63%), *Z*-caryophyllene **4** (R_t 7.11 min, 1.84%), α-humulene **5** (R_t 7.59 min, 1.77%), caryophyllene D **6** (R_t 7.98 min, 1.91%), α-bulnesene **7** (R_t 8.32 min, 1.84%), cubenol **8** (R_t 10.25 min, 0.61%), 8-heptadecene (R_t 10.47 min, 1.95%), farnesol (R_t 11.02 min, 1.91%), 4-hydroxy-3,5-dimethoxyacetophenone (R_t 11.17 min, 1.71%), palmitic acid (R_t 13.66 min, 2.61%), aesculetin dimethyl ether **9** (R_t 13.78 min, 2.28%), (*Z*) phytol (R_t 15.17 min, 0.97%) and campesterol (R_t 22.19 min, 16.04%). Fraction Fr5a afforded β-sitosterol and stigmasterol (1:1) (identified by their ^{1}H-NMR spectra).

Fraction Fr5b afforded by GC/MS benzoic acid (R_t 5.43 min, 0.38%), 4-hydroxy-3,5-dimethoxyacetophenone (R_t 13.21 min, 0.03%), coniferyl alcohol (R_t 13.27 min, 0.09%), myristic acid (R_t 13.53 min, 0.08%), 2,3,6,7-tetrahydrofarnesol (R_t 14.38 min, 0.11%), 6,10-dimethyl-2-undecanone (R_t 14.14 min, 0.14%), pentadecanoic acid (R_t 14.63 min, 0.05%), methyl palmitate (R_t 15.29 min, 0.24%), palmitic acid (R_t 15.72 min, 5.23%), heptadecanoic acid (R_t 16.68 min, 0.11%), (*Z*) phytol (R_t 17.18 min, 2.65%), stearic acid (R_t 17.66 min, 1.07%), arachidic acid (R_t 19.48 min, 0.59%) and campesterol (R_t 36.70 min, 1.44%).

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. Many 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. After that, the structure was further ascertained by comparing their mass spectral data with those available in Adams[9] or of the corresponding compound from the literature.

In addition to many fatty acids and fatty acid derivatives, about six shikimate derivatives, eight monoterpenes and six sesquiterpenes were identified from the species under investigation, *Trigonella stellate*, which has not been previously investigated for its volatile components.



Scheme 1

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المكونات الكيميائية لنبات السطيح « ترايجونيلا ستيلات »

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المستخلص. في هذا البحث ، تم جمع نبات السطيح « ترايجونيلا ستيلات » من منطقة وادي قطان ، شمال شرق جبل هازن ، حوالي ١٣٠كم جنوب الطائف بالمملكة العربية السعودية ، وفحصت المكونات الطيارة باستخدام تقنية GC/MS فنتج عن البحث تعريف حوالي اثنين وعشرين مركباً متطايراً ، بالإضافة إلى العديد من مشتقات الدهون وبيتاسيتوستيرول واستجماستيرول . هذا النوع من الترايجونيلا لم يفحص محتواه من المنتجات الطبيعية من قبل.