

CHEMICAL COMPOSITION OF THE ESSENTIAL OIL OF *TAGETES MINUTA* GROWING IN SAUDI ARABIA

**Kadriya S. EL- Deeb*, Fawkeya A. Abbas, Ahlam El Fishawy
and Jaber S. Mossa**

تم في هذا البحث تحليل الزيت الطيار المحضر بتقطير نبات تاجيتيزمينوتا لينيه الذي ينمو في المملكة العربية السعودية باستعمال تقنية كروماتوجرافيا الغاز المتصلة بمطياف الكتلة. وتبين من الدراسة أن التاجيتون (11.52%) وال-5 أوكتين-4 ون-2,7 ثنائي الميثيل (11.52%) وبروبان نيترايل وثنائي سيكلوهيكسائل (10.45%) و-2 بينين-4 ون (8.03%) تمثل المركبات الأساسية في هذا الزيت وكميات قليلة من 1-أستوكسي - بارا- مينث-3 ون (0.17%) و-9 أوكتاسيناميد (2) (0.48%) تبين وجودها في هذا الزيت وتبين أن العينة ليس لها تأثير مضاد للميكروبات أو الفطريات.

Analysis of the essential oil of *Tagetes minuta* L. growing in Saudi Arabia by gas-chromatography-mass spectroscopy (GC/MS) technique confirmed that tagetone (11.52%), 5-octyn-4-one,2,7-dimethyl (11.52%), propanedinitrile, dicyclohexyl- (10.45%) and 2-pinen-4-one (8.03%) being the main components. Lesser amounts of 1-acetoxy-*p*-menth-3-one (0.17%) and 9-octacenamide(*Z*) (0.48%) were also found in this oil. The oil did not show neither antimicrobial nor antifungal activity.

Key Word: *Tagetes minuta* L., Asteraceae, essential oil composition, antimicrobial.

Introduction

Tagetes minuta L. (Asteraceae) is a native plant in Argentina and is known as suico or chinchilla and has been used in popular as anthelmintic, diuretic, antispasmodic and to treat stomach and intestinal diseases (1). Several studies have been made on the volatile oil of *Tagetes* species. It was found that the *Tagetes* species can be unambiguously differentiated by the chemical composition of their essential oils (2-5). The volatile oil prepared from the ornamental plant "Irish Lace" (*Tagetes filifolia* Lag.) growing wild in Mexico, contains more than 95 % of esdragole (6). (*E*)-ocimene in the essential oils of *Tagetes minuta* L. (syn. *T. glandulifera* Schrank.) growing in Hungary contributes about 30-40 % and in *T. tenuifolia* Cav. (syn. *T. signata* Bartl.) about 5-10 % (5).

The main components of the essential oil of *T. patula* L. were reported to be limonene, caryophyllene, piperitone and piperitenone (7-11). Machalo *et al.* (12) characterized twenty seven compounds in the oil of *T. erecta* L. collected from Brazil. The major compounds were terpinoline (12.4 %), (*E*)-ocimene (13.1%), piperitone (20%) and limonene (11%). Indole was found among the minor compounds. The characteristic components of the essential oil of *T. lucida* Cav. are linalool, esdragol and methyl eugenol (12). (*Z*)-ocimene (43.62-45.59%) and (*E*)-ocimene (37.29-40.38 %) were the main components of *T. argentina* (13). Analysis of essential oil-secreting organs of *T. minuta* plants growing in Hungary by GC/MS revealed the presence of 6-10 and 16% eugenol from the involucre bract and hypsophyll, respectively, as well as dihydrotagetone, tagetone, (*Z*)-and(*E*)-ocimenes (9).

Reviewing the current literature, there are no previous chemical investigations of the constituents

Department of Pharmacognosy, College of Pharmacy, King Saud University, P.O. Box 2457, Riyadh 11451, Saudi Arabi.

*To whom correspondence should be addressed

Table 1: GC-MS identified constituents in the essential oil of *Tagetes minuta* L.

Peak No	Compound name	RT	M ⁺	Base peak	Major peaks	Relative percentage
1	Cyclohexene, 1-methyl-4(1-methylethenyl)	5.017	136	68	93,67,136,39	3.06
2	(E)-ocimene	5.042	136	93	79,41,105,121,136	7.11
3	3-Butene-1,2-diol,1-(2-furyl)-2,3-dimethyl	5.317	182	43	97,57,41,86,71,164,182	2.20
4	3-Oxatricyclo(4.1.1.0 ^{2,4})octane,2,7,7-trimethyl-	5.692	152	67	41,83,109,39,69,137,152	1.11
5	1-Acetoxy-p-menth-3-one	5.908	212	110	82,43,95,55,69,137,152,212	0.17
6	β-linalool	6.017	154	71	93,41,55,121,39,136,154	1.09
7	1,5-Heptadien-4-one,3,3,6-trimethyl	6.242	152	83	55, 109, 41,69,152	1.50
8	β-ocimene	6.475	136	91	136,105,79,91,39,55	1.28
9	(Z) and (E) Epoxy-ocimene	6.508	152	79	41,39,67,93,109,134,152	1.20
10	4,4-Dimethyl-2-cyclopenten-1-one	6.700	110	95	67, 110,41,27,53,81	8.03
11	Tagetone (Z)	6.850	152	95	67,109,41,152,137	11.52
12	5-Octyn-4-one,2,7-dimethyl	6.850	152	95	65,41,110,27,137,53,152	11.52
13	1,4-Pentadiene,2,3,3-trimethyl	6.967	110	95	67,41,110,39,69,96	1.23
14	Epoxylinool	7.033	170	59	43,94,68,93,111,155,170	0.67
15	2,6-Dimethyl-3,5,7-octatriene-2-ol,E	7.525	152	43	109,81,67,91,152,137	0.52
16	Cyclohexene,2-ethenyl-1,3,3-trimethyl	8.009	150	135	93,79,107,150,41,55,39	5.68
17	2-Pinen-4-one	8.008	150	135	107,150,80,91,39,55,122	8.03
18	Verbenone	8.125	150	135	107,150,80,79,41,108,55	6.73
19	Propanol,3-cyclohexylidene-2-methyl-	8.250	152	55	81,123,41,39,110,152,133	0.28
20	Limonene dioxide	8.342	168	43	55,67,107,93,109,123,153,168	0.71
21	2-Isopropyl-5-methyl-6-oxabicyclo(3.1.0)hexane-1-carboxaldehyde	8.417	168	125	43,55,69,97,83,39,168,140	2.13
22	Carvone	8.558	150	82	39,54,108,150,107,79,135	3.99
23	2-Cyclohexen-1-one,3-methyl-6-(1-methylethenyl)-,(+)	8.567	150	82	39,150,135,107,54,91,122	4.96
24	3-Methoxy-2,5,6-trimethylphenol	10.267	166	149	166,151,39,91,77,123,53	1.72
25	6-Z-2,5,5,10-Tetramethyl-undeca-2,6,9-trien-8-one	17.633	220	83	137,41,55,69,109,151,123,220	0.45
26	Piperitenone	18.008	150	150	150,107,135,91,39,109,79,149	0.55
27	Limonen-6-ol,pivalate	19.042	236	83	57,41,93,107,119,79,134,236	1.15
28	Propanedinitrile, dicyclohexyl-	19.392	230	83	55,41,39,148,81,105,230	10.45
29	9-Octadecenamide(Z)	20.775	281	59	72,41,29,83,97,114,281	0.48

of the volatile oil of *Tagetes minuta* L. growing in Saudi Arabia. Therefore analysis of this oil was performed in order to identify its constituents as well as to determine its antimicrobial activity.

Experimental

Plant Material:

Tagetes minuta L. (Asteraceae) was collected during the flowering stage in June 1999 from the Southern region of the Kingdom of Saudi Arabia. The plant material was kindly identified by Dr. Sultan Abidin, Prof. of Taxonomy, Faculty of Pharmacy, KSU.

Preparation and Analysis of the Oil:

The oil was prepared by hydrodistillation (14) of the fresh herb (100 g) for 8 hours. The oil was dried over anhydrous sodium sulfate (to give 0.3%) and kept at 4° C in sealed vials for analysis. The GC/MS analysis was carried out on Gas Chromatography Mass Spectrometer GC-17A Shimadzu, Auto Injector- AOC -201, Shimadzu, Japan, using capillary column of fused silica (30 m x 0.25 mm i.d.) coated with 5% phenyl methyl polysiloxane (DB-5). Helium ultra pure was used as carrier gas at flow rate 25 ml/min. Oven temperature was programmed at 60-200 °C at 10° C/min., the injection mode split ratio was 1: 50, column inlet program was 25.5 cm/sec and the MS detection routine was from 9-60 min. The scan of m/z was from 80-550 and the Start-Stop masses were from 40-600. Identification of the constituents was performed by aid of the computer library search (CAS No. 5989-27-5, Entry 8747, LIB# 1). Results are recorded in Table 1.

Antimicrobial Screening:

The antimicrobial screening of the oil was performed according to the general qualitative assay described by Clark *et al.* (15) using the following microorganisms: *Escherichia coli*, *Staphylococcus aureus* and *Candida albicans*, all of them are clinical isolates strains obtained from stock cultures of the Department of Microbiology, Faculty of Pharmacy, KSU.

Results and Discussions

The volatile oil of *Tagetes minuta* L. was prepared by hydrodistillation (14) of the fresh herb. The percentage of the essential oil of the fresh herb of *Tagetes minuta* L. was 0.3 % v/w. It is a yellow liquid with a characteristic odour. It exhibited RI 1.401 and specific gravity 0.688. The oil is soluble

in ether and ethanol, insoluble in water. GC/MS analysis resulted in the identification of twenty nine components (Table 1). Among these tagetone(Z) (11.52%), 5-octyn-4-one,2,7-dimethyl (11.52%), propanedinitrile, dicyclohexyl-(10.45%) and 2-pinen-4-one (8.03%) being the main components. While 1-acetoxy-*p*-menth-3-one (0.17%) and 9-octadecenamide(Z) (0.48%) were present as minors or traces. (*E*)-ocimene was found to be only (8.39 %) in contrast to the reported data in Hungarian species (30-40%). The oil did not show any antimicrobial or antifungal activities against the tested microorganisms.

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