

RESEARCH ARTICLE

Essential Oil Content and Compositions of Naturalized *Tagetes minuta* L. (Wild marigold)

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Abstract

Tagetes minuta L. known as Muster John Henry is an annual, fragrant and naturalized plant species in Turkey. The study aimed to determine essential oil content and components of *Tagetes minuta* L. from the flora of Hatay. Flower and leaf parts were hydro-distilled together in the full flowering period. Essential oils were analysed by gas chromatography-mass spectrometry. Essential oil content was found at 1.8%. Essential oil compositions that constitute 99.20% of the essential oil were revealed. Main components found as *trans*- β -ocimene 45.92% and verbenone 32.68%. *T. minuta* has wide usage in perfume, food and ornamental plant industry. Further studies should perform on the cultivation of the species for national industries.

Keywords: Mexican marigold, Asteraceae, gas chromatography

Introduction

Tagetes minuta from the sunflower (Asteracea) family is an annual and perennial herbaceous plant that contains many seconder metabolites. The species is native to North and South American countries, however it is introduced and naturalized in a wide range of climatic conditions. Turkey has 1500 ca. species from the sunflower family but *T. minuta* is not native to the flora. Turkey is intersection of three continents and mobility of commodities so that many plants introduced in the flora. *T. minuta* is one of them that naturalized in the flora of Turkey (Uludağ et al., 2017). *T. minuta* is native to mild temperate to temperate regions also could grow and cultivated around the world. In Turkey, this species exists in many grids (A7, B6, B7, B8, B9, C1, C5) near fields, orchards and path to forests (TUBIVES, 2020). Furthermore, until now *T. minuta* was not recorded in flora of Hatay.

T. minuta know as 'Wild marigold', 'Mexican marigold' and 'Mustar John Henry' in English, also known as 'Kadife çiçeği' in Turkish. The species has been used as food, perfumes, medicines, ornamentals and ethno practices (Hamayun et al., 2006). *T. minuta* both cultivated and collected from wild due to its high essential oil yield that has a usage in nutraceutical, perfumery, ornamental, pharmaceutical, agarbattis and agricultural industries (Chalchat et al., 1995). Besides that, it is regarded as a noxious weed and compete with cultivated plants in the farm (Holm et al., 1997). Allelopathic effect of *T. minuta* essential oil on *Zea mays* root has been proved (Scrivanti et al., 2003). India is the main exporters of tagetes oil and main importer is USA (Zaub, 2020).

T. minuta florets that have orange-yellow carotenoid lutein substance could be used as food additives (Timberlake and Henry, 1986). *T. minuta* has also many biological properties such as anti-oxidative, anti-inflammatory, acaricide, insecticidal, fungicidal, stomachic (Sadia et al., 2013; Karimian et al., 2014). Furthermore, essential oil has been shown to possess anti-inflammatory, tranquilizer, hypotensive, bronchodilator activities (Singh et al., 2003).

Dörtyol (Hatay), located in the Mediterranean phytogeographic region, three main vegetation types could be found (Türkmen, 2018) in the area. In Yahyali plateau of Dörtyol where the plant samples were collected has

vegetation of European-Siberian origin. In the area where Northern Amanos (Nur) Mountains situated, *Pinus brutia* and *Quercus cerris* are main tree species (Kara et al., 2010).

In the present study, essential oil content and the essential oil composition of *T. minuta* from Dörtyol district of Hatay was evaluated.

Materials and Methods

Plant Material

Aerial parts of *Tagetes minuta* L. plants were collected Yahyali plateau road, Dörtyol, Hatay (Turkey) in October. The geographic coordinates of plant were 36° 49' N: 36° 17'E, from 750m altitudes.

Isolation and Analyse of the Essential Oil

Both flower and leaves of fully flowering plants were hydro distilled for 3 hours with using Clevenger-type apparatus. Essential oil yield was calculated as the mean value from dry plant material weight, and expressed in g/100 g dry weight percentage. Essential oils were kept in amber vials at +4 °C until analysis. Essential oil components were analysed with a GC-MS (Gas Chromatography Mass Spectrometry) device Thermo Scientific ISQ Single Quadrupole. 5 µl of essential oil was diluted in 2 ml cyclohexane. For separation, TG-Wax MS (5% Phenyl Polysilphenylene-siloxane, 0.25 mm inner diameter * 60 m length, 0.25 µm film thickness) column was used. The ionization energy was calibrated as 70 eV, and the mass interval was m/z 1.2- 1200 amu. The scan mode was used as the screening more in data collection. MS transfer line temperature was 250°C, MS ionization temperature was 220°C, and whereas colon temperature was 50°C at the beginning, then it was increased up to 220°C with 3°C/min rate. The structure of each component was defined using mass spectrums (Wiley 9) with Xcalibur software. Retention indices were determined using retention times of *n-alkanes* (C8-C40) that were injected after the *T. minuta* essential oil under the same chromatographic conditions.

Results and Discussion

Essential oil from the air dried flower-leaf mix of *T. minuta* obtained by hydro distillation and was analysed by GC-MS. Essential oil yield was found as 1.8%. The essential oil composition of *T. minuta* is given in Table 1, together with their relative retention indices and with relative percentages. Main components of the essential oil were found as *trans*-β-ocimene (45.92%), verbenone (32.68%), anethole (6.67%) and dihydrocarvone (3.78%).

Previous studies on essential oils obtained four *Tagetes* species from Hungary revealed that oil ranged between 0.50-1.10% (Hethelyi et al. 1986) and main components found as follows; β-ocimenone 38.6, (*E*)-ocimenone %26.0, tagetone 10.7%, dihydrotagetone 10.6%, (*Z*)-ocimenone 5.0%. Chalchat et al. (1995), reported essential oil content and components of *T. minuta* flowers (in 8 locations) and leaves (only in one location) from Rwanda and France. In that study researchers found essential oil content between locations 0.1-1.1%. *T. minuta* flower oil mostly comprised of (*Z*)-β-ocimene and leaf oil mostly comprised of dihydrotagetone. Dihydrotagetone ranged between 2.7-49.2% while (*Z*)-β-ocimene presents 31.0-39.0%. In that study location did not found effective on essential oil content. Essential oil content of *T. minuta* collected in Bursa, Turkey during full flowering period was found as 1.81% (Baser, 1996). In the same study, main components of essential oils were found as follows; (*Z*)-β-ocimene 28.49%, dihydrotagetone 30.30% and (*E*)-tagetone 15.35%.

Table 1. Essential oil composition of *T. minuta* L. (%)

RI*	Ref. RI**	Compound Name	Area %
1098	1098	camphene	0.05
1111	1083	ethyl 2-methylbutyrate	0.34
1148	1147	sabinene	0.24
1160	1161	α -myrcene	0.05
1179	1194	phellandrene	0.30
1205	1205	limonene	2.33
1227	1229	<i>trans</i> - β -ocimene	45.92
1242	1245	1,3,7-Octatriene, 3,7-dimethyl	0.44
1301	1299	<i>o</i> -cymene	0.07
1401	1400	<i>allo</i> -ocimene	0.65
1405	1384	α -pinene-oxide	0.33
1432	1430	cyclohexanol	0.07
1533	1533	linalool	0.26
1621	1624	dihydrocarvone	3.78
1716	1701	estragole	1.13
1723	1732	α -humulene	0.17
1747	1723	verbenone	32.68
1836	1847	anethole	6.67
2084	2129	caryophyllene oxide	0.41
2125	2129	spathulenol	3.13
Total			99.20

The cultivated *T. minuta* leaf essential oil was found to be approximately 1%. The main components found as *cis*- β -ocimene (33%) along with *E*-ocimene (19%) and tagetone (16%) from Iran (Karimian et al., 2014). Essential oil of non-blooming plant leaves mostly contained dihydrotagetone (25.29-54.66%), while blooming flowers and leaves were rich in β -ocimene (11.5-45.4%) and tagetenone (11.7-32.9%) from Argentina (Chamorro et al., 2008).

Domestication of the plant was studied in India. In the study different climatic regions with different cultivation period for 1-3 harvesting in the season were used. Average essential oil content ranged between 0.25-0.70%. Main components of essential oil were ocimene (11.8-45.0%), dihydrotagetone (7.6-34.1%), tagetones (10.3-28.53%) and ocimenones (17.2-42.4%). Study showed that in winter harvests ocimene was higher while dihydrotagetone was sharply lower (Singh et al., 2003).

T. minuta essential oil mostly contains (*E*)- β -ocimene unsaturated acyclic monoterpene hydrocarbon, verbenone, a bicyclic monoterpene ketone, anethole unsaturated ether and tagetone unsaturated acyclic monoterpene ketone. Ocimene is known with the sweet herbal odor, also known as one of the main component of basil and lavender (Parker et al., 2015; Ali et al., 2015). In *Medicago truncatula*, it was found that ocimene plays an active role in indirect insect defense (Navia-Gine et al., 2009). Arimura et al. (2004) indicated that *L. japonicus* plants infested with two-spotted spider mites (*Tetranychus urticae*), coincide with increasing emissions of (*E*)- β -ocimene with regulating of LjE β OS transcripts. Kishimoto et al. (2005) indicated that volatile treatment on plants could result retardation of fungal disease (*Botrytis cinerea*) development. Another main component of *T. minuta* verbenone, mostly found in *Verbena triphylla* (Spanish verbena) and *Rosmarinus officinalis*, that has also antimicrobial activity (Santoyo et al., 2005).

The present study revealed essential oil content and components of *T. minuta* plants from Hatay, Turkey. In other countries *T. minuta* is cultivated and collected for essential oil, however it has no collection and cultivation in Turkey. Comparing the results of present study with previous researches essential oil content was found higher than the cultivated samples and the essential oil had higher β -ocimene and verbenone contents. Regarding these results, cultivation of *T. minuta* in Turkey especially in Hatay for use in different industries should be evaluated.

REFERENCES

- Ali, B., Al-Wabel, N. A., Shams, S., Ahamad, A., Khan, S. A., Anwar, F. (2015). Essential oils used in aromatherapy: A systemic review. *Asian Pacific Journal of Tropical Biomedicine*, 5(8), 601-611.
- Arimura, G., Ozawa, R., Kugimiya, S., Takabayashi, J., Bohlmann, J. (2004). Herbivore-induced defense response in a model legume. Two-spotted spider mites induce emission of (E)-beta-ocimene and transcript accumulation of (E)-beta-ocimene synthase in *Lotus japonicus*. *Plant Physiology*, 135(4), 1976–1983.
- Baser, K. H. C. (1996). Essential oil of *Tagetes minuta* L. from Turkey. *Journal of Essential Oil Research*, 8, 337-338.
- Chalchat, J. C., Garry, R. P., Muhayimana, A. (1995). Essential oil of *Tagetes minuta* from Rwanda and France: chemical composition according to harvesting, location, growth stage and part of plant extracted. *Journal of Essential Oil Research*, 7, 375-386.
- Chamorro, E. R., G. Ballerini, A. F. Sequeira, G. A. Velasco and M. S., Zalazar (2008). Chemical composition of essential oil from *Tagetes minuta* L. leaves and flowers. *Journal of Argentine Chemical Society*, 96(1-2), 80- 86.
- Hamayun, M., Hussain, F., Afzal, S., Ahmad, N. (2006). Allelopathic effect of *Cyperus rotundus* and *Echinochloa crus-galli* on seed germination, and plumule and radical growth in maize (*Zea mays* L.). *Pakistan Journal of Weed Science Research*, 11, 81-84.
- Hethelyi, E., Danos, B., Tetenyi, P., Koczka, I. (1986). GC MS analysis of the essential oils of four *Tagetes* species and the anti-microbial activity of *Tagetes minuta*. *Flavour and Fragrance Journal*, 1,169-173.
- Holm, L. D. J., Holm, E., Pancho, J., Herberger, J. (1997). *Tagetes minuta* L. Asteraceae (Compositae) Family. In *World weeds: Natural Histories and Distribution* (pp. 822-827). John Wiley & Sons, Inc., New York.
- Kara, R., Ezer, T., Düzenli, A. (2010). The Epilithic (Serpentine) Bryophytes of the Northern Amanos (Nur) Mountains. *Biyoloji Bilimleri Araştırma Dergisi*, 3(2), 129-136.
- Karimian, P., Kavooosi, G., Amirghofran, Z. (2014). Anti-oxidative and anti-inflammatory effects of *Tagetes minuta* essential oil in activated macrophages. *Asian Pacific Journal of Tropical Biomedicine*, 4(3), 219-227.
- Kishimoto, K., Matsui, K., Ozawa, R., Takabayashi, J. (2005). Volatile C6-aldehydes and Allo-ocimene activate defense genes and induce resistance against *Botrytis cinerea* in *Arabidopsis thaliana*. *Plant & Cell Physiology*, 46(7), 1093–1102.
- Navia-Giné, W. G., Yuan, J. S., Mauromoustakos, A., Murphy, J. B., Chen, F., Korth, K. L. (2009). *Medicago truncatula* (E)- β -ocimene synthase is induced by insect herbivory with corresponding increases in emission of volatile ocimene. *Plant Physiology and Biochemistry*, 47(5), 416-425.
- Parker, J. K. (2015). Introduction to Aroma Compounds in Foods: In *Flavour Development, Analysis and Perception in Food and Beverages*, eds. J. K., Parker, S., Elmore and L., Methven. United Kingdom: Woodhead Publishing.
- Scrivanti, L. R., Zunino, M. R., Zygodlo, J. A. (2003). *Tagetes minuta* and *Schinus areira* essential oils as allelopathic agents. *Biochemical Systematics and Ecology*, 31(6), 563-572.
- Singh, V., Singh, B., Kaul, V. K. (2003). Domestication of wild marigold (*Tagetes minuta* L.) as a potential economic crop in western Himalaya and north Indian plains. *Economic Botany*, 57, 535-544.

Sadia, S., Khalid, S., Qureshi, R., Bajwa, A. A. (2013). *Tagetes minuta* L., a useful underutilized plant of family Asteraceae: a review. *Pakistan Journal of Weed Science Research*, 19(2), 179-189.

Santoyo, S., Cavero, S., Jaime, L., Ibáñez, E., Señoráns, F., Reglero, G. (2005). Chemical composition and antimicrobial activity of *Rosmarinus officinalis* L. essential oil obtained via supercritical fluid extraction. *Journal of Food Protection*, 68, 790-795.

Timberlake, C. F., Henry, B. S., 1986. Plant pigments as natural food colours. *Endeavour Journal*, 10, 31–36.

TUBIVES (2020, February 10). http://194.27.225.161/yasin/tubives/index.php?sayfa=1&tax_id=4681

Türkmen, N. (2018). The plant diversity and conservation status of the Taurus and Amanos mountains in the Eastern Mediterranean Region of Turkey. *Biodiversity Journal*, 9(4), 385-394.

Uludağ, A., Aksoy, N., Yazlık, A., Arslan, Z. F., Yazmış, E., Üremiş, I., Cossu, T. A., Groom, Q., Pergl, J., Pyšek, P., Brundu, G. (2017). Alien flora of Turkey: checklist, taxonomic composition and ecological attributes. *NeoBiota*, 35, 61–85. <https://doi.org/10.3897/neobiota.35.12460>.

Zauba (2020, February 10). *Tagetes minuta* export data. Retrieved from <https://www.zauba.com/export-OILS+TAGETES+OIL-hs-code.html>