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The Essential Oil Composition of *Tanacetum densum* (Labill.) Heywood ssp. *eginense* Heywood from Turkey

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Abstract: Water-distilled essential oils from aerial parts of *Tanacetum densum* (L.) Heywood ssp. *eginense* Heywood, from Turkey was analysed by GC and GC-MS. *T. densum* ssp. *eginense* flower, stem and leaf oils were characterized with camphor (30.9% , 25.7%, 27.7%), 1,8-Cineole (12.4% flower oil), camphene (10.6%, %7.0, flower and leaf oils), bornyl acetate, (9.4%, 11.8%, stem and leaf oils), *α*-pinene (7.0%, %5.3, flower and leaf oils), borneol (5.1%, 5.2%, stem and leaf oils), neodihydrocarveol (5.1%, flower oil). An unidentified compound was also present in flower, stem and leaf oils (11.5%, 27.2%, 20.5%). A comparison is done with the previous investigations on the other subspecies of *T. densum* and the differences were investigated. Flower and stem oils did not show any significant activity to the tested microorganisms when compared to positive control chloramphenicol. Flower and stem oils both showed cytotoxicity to *Vibrio fischeri.*

Keywords: *Tanacetum densum* ssp. *eginense;* Astereceae; Essential oil; antibacterial activity; *Vibrio fischeri* cytotoxic activity; camphor; 1,8-cineole; camphene; bornyl acetate; α-pinene; borneol; neodihydrocarveol.

1. Plant Source

Tanacetum densum is an endemic species of Turkey which is represented by four subspecies. *T. densum* ssp. *eginense* and ssp. *amani* plants have very similar morphological properties and they could only be separated from each other by the decoloration on the edge of their phyllaries [1].

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2. Previous Studies

Previously chemistry of *T. densum* ssp. *amani*, ssp. *sivasicum* and ssp. *eginense* were reported. New sesquiterpene lactones sivasinolide [2], 1*β*,4*α*,6*α*-trihydroxyeudesm-11-en-8*α*,12-olide [3] and new farnesols 11-hydroxy-5,14-diacetoxy-9,10-dehydrofarnesol acetate, 10-hydroxy-5,14-diacetoxy-11,12-dehydrofarnesol acetate [4] were isolated together with known sesquiterpene lactones, flavonoids, triterpenes and phenolics from *T. densum* (Labill.) Heywood ssp. *sivasicum* Hub.-Mor. et Grierson and ssp. *amani* [2-5]. Previous investigation on chemistry of *T. densum* ssp. *eginense* reported isolation of new eudesmane type sesquiterpene lactone eginense together with known sesquiterpene lactones, farnesols, flavonoids and triterpenes [6]. Also essential oil composition of all subspecies [7,8,10] except *T. densum* ssp. *eginense* were previously investigated. Comparison of previously reported main components of *T. densum* essential oils together with present work is given in Table 1. According to three previous reports from different research groups presents variation in the essential oil composition of *T. densum* ssp. *amani* [7,10,11].

To the best of our knowledge there is no report on the essential oil composition of *T. densum* ssp. *eginense.* As a part of our phytochemical and biological investigation of *Tanacetum* species [8,9,11-13] here we report on the essential oil composition of endemic *T. densum* ssp. *eginense* from Turkey. As indicated before morphologically very similar plants *T. densum* ssp. *eginense* and ssp. *amani* were compared with each other according to their essential oil compositions. Comparison of essential oil components of all previously investigated *T. densum* subspecies was also given.

3. Present Study

Plant Material: Plant materials were collected during the flowering period in july 2002 from the south face of the Tecer Mountain in vicinity of Sivas city at 1900 m altitude. Voucher specimens have been deposited at the Herbarium of the Faculty of Science, Istanbul University (Voucher no. ISTE 80538). Plant materials were identified by Dr. Kerim Alpınar.

Isolation of the Essential Oils: Flowers and stems (100g each) of the plants were subjected to hydrodistillation for 4 h using a Clevenger-type apparatus to produce the essential oils. Essential oil yields of flower, stem and leaves oils are 0.35, 0.25 and 0.45 respectively.

Gas Chromatography-Mass Spectrometry Analysis: Results of the analysis were given in Table 1. Method employed in the analysis was given in supporting information **S1**.

Table 1. Essential oil composition of *T. densum* ssp. *eginense* oils. (*continued overleaf*)

RRI	Compound	A (%)	B (%)	C (%)
1014	Tricyclene	0.7	0.2	0.4
1032	*α*-Pinene	7.0	3.1	5.3
1076	Camphene	10.6	4.4	7.0
1093	Hexanal	0.2	t	-
1118	*β*-Pinene	1.6	0.8	1.3
1132	Sabinene	0.1	t	0.1
1188	*α*-Terpinene	0.2	t	t
1195	Dehydro-1,8-cineole	t	-	-
1203	Limonene	0.8	1.0	1.1
1213	1,8-Cineole	12.4	3.2	2.9
1244	Amyl furan (2-pentyl furan)	t	t	t
1255	*γ*-Terpinene	0.3	0.1	0.1
1265	5-Methyl-3-heptanone	-	t	t
1280	*p*-Cymene	0.6	0.3	0.3
1285	Isoamyl isovalerate	0.1	-	-
1290	Terpinolene	0.1	t	-
1296	Octanal	0.1	-	-
1345	3-Octylacetate	-	-	t
1348	6-Methyl-5-hepten-2-one	-	t	-

1360	Hexanol	-	t	-
1386	1-Octenylacetate	-	t	-
1393	3-Octanol	-	0.1	t
1400	Nonanal	0.1	t	-
1452	1-Octen-3-ol	0.1	0.1	0.2
1463	1-Heptanol	t	0.1	0.1
1474	*trans*-Sabinene hydrate	0.1	0.2	0.1
1499	*α*-Campholene aldehyde	0.1	0.2	0.2
1522	Chrysanthenone	-	0.1	t
1532	Camphor	30.9	25.7	27.7
1544	Cyprene	-	0.3	-
1556	*cis*-Sabinenehydrate	0.1	0.2	0.2
1568	1-Methyl-4-acetylcyclohex-1-ene	0.3	0.7	0.8
1586	Pinocarvone	0.6	0.1	0.5
1590	Bornylacetate	4.3	9.4	11.8
1611	Terpinen-4-ol	-	0.4	0.3
1624	*trans*-dihydrocarvone	-	0.5	0.5
1637	Unknown	11.5	27.2	20.5
1639	*trans*-*p*-Mentha-2,8-dien-1-ol	-	0.4	0.3
1670	*trans*-Pinocarveol	0.7	0.7	0.6
1678	*cis*-*p*-Mentha-2,8-dien-1-ol	0.2	0.2	0.1
1683	*trans*-Verbenol	0.1	0.3	0.2
1704	Myrtenyl acetate	-	-	0.1
1706	*α*-Terpineol	0.2	-	-
1719	Borneol	3.6	5.1	5.2
1726	Germacrene-D	0.1	0.1	0.2
1732	Neodihydro carveol	5.1	3.5	2.4
1747	*trans*-Carvylacetate	1.0	1.5	1.2
1751	Carvone	0.3	0.6	0.4
1757	Dihydrocarveol	0.1	-	-
1764	*cis*-Chrysanthenol	-	-	0.3
1782	*cis*-Carvylacetate	t	t	t
1804	Myrtenol	0.2	0.2	0.2
1811	*trans*-*p*-Mentha-1(7), 8-dien-2-ol	0.2	0.4	0.2
1827	(*E*,*E*)-2,4-Decadienal	-	t	t
1845	*trans*-Carveol	0.5	0.6	0.4
1865	Isopiperitenone	-	-	t
1868	(*E*)-Geranylacetone	-	0.1	-
1882	*cis*-Carveol	0.1	0.1	0.1
1896	*cis*-*p*-Mentha-1(7), 8-dien-2-ol	0.3	0.4	0.2
2008	Caryophyllene oxide	-	t	0.2
2088	1-*epi*-Cubenol	-	-	0.1
2144	Spathulenol	0.1	-	0.1
2179	Tetradecanol	-	t	-
2209	T-Muurolol	t	-	0.1
2232	*α*-Bisabolol	-	t	0.1
2316	Caryophylla-2(12), 6(13)-diene-5 *β*-ol	-	-	0.1
2324	Caryophylla-2(12), 6(13)-diene-5 *α*-ol	-	t	0.1
2384	1-Hexadecanol	0.1	0.6	0.5
2438	Kaur-16-ene	-	0.2	0.2
2500	Pentacosane	-	0.1	0.1
2700	Heptacosane	-	-	0.2
2900	Nonacosane	-	0.2	-
Monoterpene hydrocarbons		**22.0**	**9.9**	**15.6**
Oxygenated monoterpenes		**61.1**	**54.1**	**56.1**
Sesquiterpene hydrocarbons		0.1	0.4	0.2
Oxygenated sesquiterpenes		0.1	-	0.8
Diterpenes		-	0.2	0.2

Others	1.0	1.9	2.0
Total identified compounds	**84.3**	**66.5**	**74.8**

Compounds were given according to their retention times in HP-Innowax coloumn. Relative percents of the compounds were given in the table. RRI: Relative Retention Indices; t: Trace (<0.1%); **A**: *T. densum* ssp. *eginense* – flower oil; **B**: *T. densum* ssp. *eginense* – stem oil; **C**: *T. densum* ssp. *eginense* – basal leaves oil. **Unknown**: EIMS, 70 eV, m/z (rel. int.): 154(4), 136(93), 107(100), 93(78), 79(67), 67(26), 55(20), 43(85).

Antibacterial Activity Test: Method employed in the tests was given in supporting information **S2**. Results of the antibacterial tests were given in Table 3 which was given in supporting information **S5**.
Vibrio Fischeri Toxicity: Method employed in the tests was given in supporting information **S3**. Results of the *Vibrio fischeri* toxicity tests were given in Table 3 which was given in supporting information **S5**.

Water-distilled essential oils from herbal parts of *T.densum* ssp. *eginense* were analysed by GC and GC-MS. The compounds identified with their percentages could be seen in Table 1. Forty-five, fifty-seven and fifty-seven compounds were identified in the oils of *T. densum* ssp. *eginense*, representing 84.3%, (flower), 66.5% (stem), and 74.9% (leaf), of the oils. Camphor (30.9%), (25.7%) and (27.7%) was the main constituent of the flower, stem, and basal leaf oil respectively. 1,8-Cineole (12.4%), camphene (10.6%), *α*-pinene (7.0%), neodihydrocarveol (5.1%) were also present in the flower oil. Bornylacetate (9.4% - stems; 11.8% - leaf), borneol (5.1% - stem; 5.2% - leaf), camphene (7% - leaf) and *α*-pinene (5.3% - leaf) were also present in the oils from stems and basal leaves. An unknown compound was observed in all of the oils with high amounts (11.5%, 27.2%, 20.5% for flower, stem and basal leaf oils respectively). Mass spectra of the compound suggested that this compound could be an oxygenated monoterpene. However molecular ion peak was not clear in EIMS spectra. Flower and stem oils were tested for their antibacterial activity against selection of bacteria. None of the tested oils showed any significant activity when compared with positive control chloramphenicol. Also the same oils were tested for their general toxicity with *Vibrio fischeri* cytotoxicity assay and both oils showed toxicity to *Vibrio fischeri* when compared with positive control Vitamin C. Essential oil composition of morphologically very similar plant *T. densum* ssp. *amani* [1] was reported in the literature [7,10,11]. Variation in the essential oil composition of *T. densum* ssp. *amani* in these reports could clearly be seen in Table 2 which is given in supporting information **S4**. Previously we also reported essential oils of this plant with high content of *α*-pinene, *β*-pinene, 1,8-cineole, *p*-cymene and lavandulyl acetate unlike other reports [11]. Previous investigations on *T. densum* ssp. *amani* reported main components *β*-patchoulene, endoborneol and (+)-*epi*-bicyclosesquiphellandrene [7,10] these compounds were not present in *T. densum* ssp. *eginense* oil. All of the *T. densum* subspecies have 1,8-cineole and camphor in high amounts except *T. densum* (Labill.) Heywood ssp. *laxum* Grierson which does not contain camphor and have 1,8-cineole in small amounts [10]. Similarly *T. densum* ssp. *amani* oils reported in two different literatures contains camphor in very small amounts and 1,8-cineole in high amounts [10,11]. *T. densum* oils were characterized with bornane type and *p*-menthane type monoterpenes except *T. densum* ssp. *laxum* which is reported to be rich in sesquiterpenes [10]. Additionally *T. densum* ssp. *amani* oil previously reported by us contains pinane type monoterpenes in high amounts unlike the rest of the *T. densum* oils [11]. In this research essential oil composition of endemic *T. densum* ssp. *eginense* flower, stem and basal leaf oils from Sivas-Turkey were investigated for the first time. Camphor and 1,8-cineole compounds were observed in main components as expected like the most of the previous investigations on *T. densum* essential oils. However *T. densum* ssp. *eginense* oil differed from the other subspecies with the high content of camphene and bornyl acetate. Camphene and bornyl acetate were not reported higher than 3% in previous reports for *T. densum* essential oils [7,8,10,11]. These differences encountered in the essential oil compositions could be related to many parameters such as the plant parts used in the analysis, different collection times, methods and instruments used in analysis, climatic and ecological factors. Flower and stem oils of *T. densum* ssp. *eginense* did not showed any significant antibacterial activity against tested microorganisms however both oils showed toxicity to *Vibrio fischeri* which is used to evaluate general toxicity of the oils.

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Supporting Information

Supporting Information accompanies this paper on http://www.acgpubs.org/RNP

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