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Essential Oil Composition of *Thymus eriocalyx* (Ronniger) Jalas from Turkey

F. ZEHRA KUCUKBAY* and EBRU KUYUMCU Division of Analytical Chemistry, Department of Basic Pharmaceutical Sciences, Faculty of Pharmacy, Inonu University, Malatya-44280, Turkey E-mail: zkucukbay@inonu.edu.tr

Hydrodistilled volatile oil from the aerial parts of *Thymus eriocalyx* (Ronniger) Jalas was analyzed by a combination of GC and GC-MS. Seventy compounds were identified, representing 98.54 % of the oil. The major components were found to be thymol (47.79 %), β -caryophyllene (11.98 %), 1,8-cineole (9.88 %), borneol (3.88 %), *p*-cymene (3.02 %), carvacrol methyl ether (2.99 %), γ -terpinene (2.97 %) and germacrene D (2.83 %).

Key Words: *Thymus eriocalyx*, GC-MS, Essential oil composition, 1,8-Cineole, Thymol, β -Caryophyllene.

INTRODUCTION

Turkey is regarded as an important gene-centre for the family Lamiaceae. The family is represented by 45 genera, 546 species and 730 taxa in Turkey. The rate of endemism in the family is 42.2 $\%^1$. Among the aromatic plants belonging to family Lamiaceae, the genus *Thymus* is noteworthy for the numerous species and varieties of wild-growing plants² and distributed in an area stretching from Mediterranean region to Europe, West Asia, North Africa, Canarian Island and North-West of Gronland Island. In Turkey it is represented by 39 species with 64 taxa, 24 of which endemic^{3,4}. Several *Thymus* species are locally known as "kekik" or "tas kekik" and their dried herbal parts are used in herbal tea, condiment and folk medicine⁵. The main constituent among them is *Thymus vulgaris* L. (thyme or garden thyme), a well-known aromatic and medicinal plant, has an increasing importance in horticulture⁶. This species was used by the ancient Egyptians in embalming. *Thymus* oils were used, alongwith glove, lemon and chamomile essential oils, as a disinfectant and antiseptic in hospitals until the beginning of the 20th century⁷. At present, the essential oils of many Thymus species are widely used as flavouring agents in food processing and many pharmacological preparations and particularly thyme oil is still among the world's top 10 essential oils⁸.

There are many reports of the essential oil composition of *Thymus* species in the literature. The main component of the oils of many *Thymus* species is thymol or carvacrol. The essential oil compositions of *T. kotschyanus* (with 41.4 % carvacrol and 19.6 % thymol)^{9,10}, *T. pubescens* (with 48.8 % carvacrol and 13.9 % thymol)¹¹,

3616 Kucukbay et al.

T. eigii (with 30.6 % thymol and 26.1 % carvacrol)¹², *T. migricus* (with 44.2 % thymol and 36.5 % carvacrol)¹³ have been reported previously.

This constitutes that 85 % of the *Thymus* taxa of Turkey have been investigated for essential oil. The survey has revealed that 24 *Thymus* taxa contained thymol and 11 taxa contained carvacrol as main constituents¹⁴. To the best of our knowledge, there is no report on the chemical composition of *Thymus eriocalyx* growing in Turkey, so the present paper reports the volatile constituents of the essential oil of *Thymus eriocalyx* (Ronniger) Jalas from Turkey.

EXPERIMENTAL

The plant material of study was collected from Van province, Turkey in June 2008. The voucher specimens have been deposited in the Herbarium of Inonu University (INU) in Malatya, Turkey (INU-Collector No.: 2682).

Extraction of the essential oil: Air-dried aerial parts of plants were hydrodistilled for 3 h using Clevenger-type apparatus. The plant was immersed in water and heated to boiling, after which the essential oil was evaporated together with water vapour and finally collected in a condenser. The distillate was isolated and dried over anhy-drous sodium sulphate. The oil was stored at 4 °C until analysis by GC and GC/MS. The percentage yield (%) of the oil calculated on a moisture-free basis was 0.51 % for TA2682 (V/W).

Gas chromatography (GC) and gas chromatography/mass spectrometry (GC/MS): The essential oils were analyzed by GC and GC/MS. GC which were carried out using an Agilent Technologies 6890N Network system. HP-Innowax column (60 m \times 0.25 mm i.d., 0.25 µm film thickness) used with helium as carrier gas. The oven temperature was kept at 60 °C for 10 min and increased up to 220 °C at a rate of 4 °C min and then kept constant at 220 °C for 10 min and increased up to 240 °C at a rate of 1 °C and then kept constant at 240 °C for 10 min. Split flow was adjusted at 84.9 mL/min. The split ratio was adjusted to 50:1. The injector and flame ionization detection (FID) detector temperatures were 250 °C.

GC/MS analysis was conducted using an Agilent technologies 5973 inert mass selective detector (Agilent G3180B Two-Ways Splitters with make up gas) system. The same column and operational conditions as in GC were applied. Helium was used as carrier gas. MS were taken at 70 eV. The mass range was between m/z 10 and 425. A library search was carried out using Wiley 7n GC/MS library, Adams library and Nist 05 library. Relative percentage amounts of the separated compounds were calculated from FID chromatograms. N-Alkanes were used as reference points in the calculation of relative retention indices (RRI).

RESULTS AND DISCUSSION

Distilled essential oils from aerial parts of *T. eriocalyx* in Turkey have been analyzed by means of GC and GC-MS. The resulting main components of the oil are shown in Table-1. The essential oil yield of *T. eriocalyx* was 0.51 %. Seventy

Vol. 22, No. 5 (2010)

compounds were identified, representing 98.54 % of the oil. The major components were found to be thymol (47.79 %), β -caryophyllene (11.98 %), 1,8-cineole (9.88 %), borneol (3.88 %), *p*-cymene (3.02 %), carvacrol methyl ether (2.99 %), γ -terpinene (2.97 %) and germacrene D (2.83 %).

Although, the essential oil composition of T. eriocalyx from different localities of Iran have also been studied^{15,16}. There are differences in the yield and constituents of the oils. It is essential to point out that geographical/environmental factors strongly influence the chemical composition of the essential oil. The abundance of thymol in the essential oils of T. eriocalyx makes them similar to those obtained in all the previous studies from Iranian species. Although there are some differences between the essential oil compositions of samples of each country. In present study together with thymol, a high amount of β -caryophyllene and 1,8-cineole were found, instead of linalool. In contrast to the report also concerned the analysis of the essential of T. eriocalyx growing in Iran^{15,16}, present study on the oil of T. eriocalyx of Turkish origin showed quantitative differences. Present sample contained linalool, α -terpineol and carvacrol in minor amounts and it was devoid of α -terpinyl acetate which constitute the main compounds in the oils obtained from several Iranian species. In addition to, the Iranian species contained carvacrol methyl ether (0.3-0.7 %) in low concentration and germacrene D was not present, which appeared as major constituents in the oils obtained from present study.

Comparison of the major components of *T. eriocalyx* oil with those of Iranian species, showed the highest amount of thymol in this oil (Table-1).

RRI	Compounds	Composition (%)
1020	α-Pinene	0.13
1023	α-Thujene	tr
1067	Camphene	0.05
1117	β-Pinene	0.05
1135	Sabinene	tr
1191	Myrecene	0.25
1212	α-Terpinene	0.20
1237	Limonene	0.24
1250	1,8-Cineole	9.88
1264	E-2-Hexenal	tr
1298	γ-Terpinene	2.97
1304	E-β-Ocimene	0.07
1307	3-Octanone	0.74
1329	<i>p</i> -Cymene	3.02
1341	α-Terpinolene	0.05
1380	3-Hexen-1-ol acetate	tr
1458	3-Octanol	0.29
1463	Nonanal	tr
1487	Thujol	0.05

TABLE-1

3618 Kucukbay et al.

Asian	T	Chom
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1509	3,4-Dimethylstyrene	tr
1516	1-Octen-3-ol	0.40
1527	α-Cubebene	tr
1529	cis-3-Hexenyl butyrate	tr
1531	trans-Sabinene hyrate	0.68
1540	cis-3-Hexenyl-2-methylbutanoate	tr
1554	cis-3-Hexenyl isovalerate	tr
1561	α-Copaene	0.09
1581	β-Bourbonene	tr
1587	Camphor	1.97
1603	β-Cubebene	tr
1606	Linalool	0.13
1608	cis-Sabinene hyrate	0.29
1615	Octanol	tr
1637	β-Ylangene	0.15
1644	Endobornyl acetate	0.40
1653	Thymol methyl ether	0.09
1655	β-Cubebene	0.09
1662	β-Caryophyllene	11.98
1663	Carvacrol methyl ether	2.99
1668	cis-Dihydrocarvone	0.19
1687	trans-Dihydrocarvone	0.05
1692	δ-Muurolene	0.15
1705	trans-Pinocarveol	0.16
1709	E-β-Farnesene	tr
1715	δ-Terpineol	0.31
1719	α -Humulene	0.63
1723	Berbenol = $trans$ -Verbenol	0.15
1733	v-Muurolene	0.24
1737	α-Terpipeol	1.08
1743	Borneol	3.88
1754	Germacrene D	2.83
1755	α -Amorphene	0.08
1762	ß Bissholene	1.00
1788	p-Disabolelle S Cadinana	0.29
1791	o-Caumene	0.17
1795	γ-Cadinene	tr
1812	p-Sesquiphellandrene	0.06
1815	Myrtenoi	0.05
1846	α -Cadinene	0.08
1852	cis-Calamene	0.13
1955	<i>p</i> -Cymen-8-01	0.09
1957	Caryophyllene oxide	0.05
1962	2-isopropyidenzaidenyde	0.55
2007	Cubanal	0.12
2030	Cuminol	0.05
2049	Spathulenol	0.21
2072	Fugenol	0.13
2083	Thymol	47.79
2098	Carvacrol	0.21
2111	Carvacion Q-Cadinol	0.20
	u-Caumon	98.54

RRI, relative retention indices; tr, trace (< 0.05 %).

Vol. 22, No. 5 (2010)

The mass spectrums of the major components of the *T. eriocalyx* were given in Figs. 1-5 with NIST library results for the comparison purposes.



Fig. 1. Mass spectrum of 1,8-cineole (top NIST library, bottom the sample spectrum)



Fig. 2. Mass spectrum of *p*-cymene (top NIST library, bottom the sample spectrum)



Fig. 3. Mass spectrum of β -caryophyllene (top NIST library, bottom the sample spectrum)

Asian J. Chem.



Fig. 4. Mass spectrum of borneol (top NIST library, bottom the sample spectrum)



Fig. 5. Mass spectrum of thymol (top NIST library, bottom the sample spectrum)

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REFERENCES

- 1. G. Esen, A.D. Azaz, M. Kurkcuoglu, K.H.C. Baser and A. Tinmaz, *Flav. Fragr. J.*, **22**, 371 (2007).
- 2. A.D. Azaz, H.A. Irtem, M. Kurkcuoglu and K.H.C. Baser, Z. Naturforsch., 59c, 75 (2004).
- 3. P.H. Davis, Flora of Turkey and the East Aegean Islands, Vol. 7. Edinburgh University Press, Edinburgh, pp. 349-382 (1982).
- 4. K.H.C. Baser, M. Vural, G. Tumen, H. Akyakin and F. Satil, Turk. J. Bot., 19, 491 (1995).
- K.H.C. Baser, Essential Oils from Aromatic Plants which are Used as Herbal Tea in Turkey, In: Flavours, Fragrance and Essential Oils, Proceedings of the 13th International Congress of Flavours, Fragrances and Essential Oils, AREP Publications, Istanbul, Turkey, pp. 67-79 (1995).
- 6. M.V.B. Reddy, P. Angers, A. Gosselin and J. Arul, Phytochemistry, 47, 1515 (1988).

Vol. 22, No. 5 (2010)

- 7. D. Ryman, Aromatherapy: The Encyclopaedia of Plants and Oils and How They Help You. Piatkus: London, UK, pp. 163-165 (1992).
- 8. E. Stahl-Biskup, J. Essent. Oil Res., 3, 61 (1991).
- 9. P.H. Davis, Flora of Turkey and the East Aegean Islands, Vol. 10, Edinburgh University Press, Edinburgh, p. 209 (1988).
- 10. G. Tumen, N. Tabanca, T. Ozek, K.H.C. Baser and G. Tumen, *Khim. Prir. Soedin.*, pp. 55-60 (1995).
- 11. F. Mericli, J. Nat. Prod., 49, 942 (1986).
- 12. B. Tepe, D. Daferera, M. Sokmen, M. Polissiou and A. Sokmen, *J. Agric. Food Chem.*, **52**, 1132 (2004).
- 13. K.H.C. Baser, B. Demirci, N. Kirimer, F. Satil and G. Tumen, Flav. Fragr. J., 17, 41 (2002).
- 14. K.H.C. Baser, Pure Appl. Chem., 74, 527 (2002).
- 15. R. Kalvandi, F. Sefidkon, M. Atri and M. Mirza, Flav. Fragr. J., 19, 341 (2004).
- 16. F. Sefidkon, R. Kalvandi, M. Atri and M. Barazandeh, Flav. Fragr. J., 20, 521 (2005).

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Contact:

Prof. Vesna B. Miskovic-Stankovic, University of Belgrade, Faculty of Technology and Metallurgy, Karnegijeva 4, 11000, Belgrade, Serbia. Tel: +381-11-330-3488, Fax: +381-11-337-0387, E-mail:vesna@tmf.bg.ac.rs Web site: http://www.rse-see.net/