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Essential oils of *Mentha* species from Marmara region of Turkey

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The genus *Mentha* L. (Lamiaceae) is represented in the world by 62 taxa including 18 species and 11 hybrids. In Turkey, 15 taxa exist. In the course of the present work, 90 samples belonging to 11 *Mentha* taxa collected from the Marmara region of Turkey in north western Turkey were hydrodistilled to obtain essential oils, which were then subjected to gas chromatographic (GC) and GC/mass spectrometric (GC/MS) analyses. Fifty-six percent of the samples contained 3-oxo-1,2-epoxy type compounds (piperitenone oxide, *cis*- and *trans*-piperitone oxide) as the main constituents in their oils, whereas 25% contained 3-oxo compounds (menthone, isomenthone, pulegone, menthofuran, piperitone, piperitenone and isopulegol). Ten percent of the samples comprised 2-oxo compounds such as carvone, *cis*- and *trans*-dihydrocarvone; 2% contained 1,8-cineole and 1% contained *trans*-sabinene hydrate as the main constituent in the oils; 5% belonged to an unknown category found in the five oils of *Mentha longifolia*.

Keywords: *Mentha pulegium*; *Mentha aquatica*; *Mentha* × *dumetorum*; *Mentha spicata*; *Mentha suaveolens*; *Mentha longifolia*; *Mentha* × *rotundifolia*; *Mentha* × *villosa-nervata*; Lamiaceae; essential oil composition

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Introduction

The genus *Mentha* L. (Lamiaceae), which is represented in the world by 62 taxa belonging to 18 species and 11 hybrids, comprises five sections within the Mentheae tribe of the subfamily Nepetoidae (1–9). Fifteen *Mentha* taxa are distributed in Turkey including six hybrids; 11 taxa including four hybrids are known to grow in the study region. In the flora of Turkey, the genus *Mentha* is divided into two sections (Table 1).

The following taxa were collected at flowering stage from the Marmara region: *Mentha pulegium* L., *Mentha aquatica* L., *Mentha* × *dumetorum* Schultes, *Mentha longifolia* (L.) Hudson subsp. *longifolia*, *M. longifolia* (L.) Hudson subsp. *typhoides* (Briq.) Harley var. *typhoides*, *Mentha spicata* L. subsp. *spicata*, *M. spicata* subsp. t*omentosa, Mentha suaveolens* Ehrh., *M.* × *villoso-nervata* Opiz, *M.* × *rotundifolia* (L.) Hudson.

Mint oils and their main constituents such as menthol, carvone, pulegone, menthone, etc. are used in perfumery, cosmetics, pharmaceutical and food industries as active constituents or as flavor or fragrance mainly as an ingredient in toothpastes, mouth washes, cigarettes, alcoholic drinks, etc. (10, 11).

Mentha species are used in Turkish folk medicine. *Mentha longifolia* known in North Anatolia as 'anik' or 'kinzi' is used as herbal tea for stomach ache, as carminative and for alleviating nervous disorders, and externally for inflammations in ointments. The plant is also known as an antiseptic and antitussive. It has been reportedly used in Java against cholera and headache (12, 13). *Mentha pulegium* known as 'Yarpuz' or 'Filiskin' is used as flavoring in foods and as a fragrance in cosmetics (10, 12, 14). During the Ottoman period, its oil used to be produced in cottage industries and sold as 'Filiskin oil' (15, 16).

Spearmint oil is produced from herbal parts of *M. spicata*, locally known is 'Bahçe nanesi (Garden mint)' or *M.* × *villoso-nervata*, a hybrid of *M. spicata* and *M. longifolia*. It is used as flavoring in foods, chewing gums, toothpastes and other oral hygiene preparations (10, 12, 14).

Previous work on Turkish mints is very scarce. Başer et al. reported on essential oils of *Mentha* species from North Anatolia (17). Other earlier works include references (18–22). Palynological studies on Turkish mints have also been published (23, 24).

A comprehensive work on mint oils appeared in 2007 (25–27).

Experimental

Plant material

Aerial parts of *Mentha* species were collected from different localities of Turkey (Table 2).

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Table 1. *Mentha* taxa recorded in Turkey.

Pulegium (Miller) DC section	
	1. *M. pulegium* L.
Mentha L. section	
Verticillatae group	
	2. *M. arvensis* L.
Capitatae group	
	3. *M. aquatica* L.
	4. *M.* × *piperita* L. (*M. aquatica* × *M. spicata*)
	5. *M.* × *piperita* L. nm. *citrata* (Ehrh.) Briq. (*M. citrata* Ehrh.) (*M. aquatica L.* var. *citrata* (Ehrh.) Fresen)
	6. *M.* × *dumetorum* Schultes (*M. aquatica* × *M. longifolia*)
Spicatae group	
	7. *M. spicata* L. subsp. *spicata*
	8. *M. spicata* L. subsp. *tomentosa* (Briq.) Harley
	9. *M. suaveolens* Ehrh.
	10. *M. longifolia* (L.) Hudson subsp. longifolia
	11. *M. longifolia* (L.) Hudson subsp. *typhoides* (Briq.) Harley var. *typhoides*
	12. *M. longifolia* (L.) Hudson subsp. *typhoides* (Briq.) Harley var. *calliantha* (Stapf) Briq.
	13. *M. longifolia* (L.) Hudson subsp. *noeana* (Boiss. ex Briq.) Briq.
	14. *M.* × *villoso-nervata* Opiz (*M. longifolia* × *M. spicata*)
	15. *M.* × *rotundifolia* (L.) Hudson (*M. longifolia* × *M. suaveolens*)

Voucher specimens of all *Mentha* species were deposited at Bursa Uludag University Herbarium (BULU).

Isolation of the essential oil

Dried aerial parts of the plant material (10–100 g) were subjected to hydrodistillation for 3 hours using a Clevenger-type apparatus to produce an oil.

Essential oil analysis

GC/MS conditions

The gas chromatographic/mass spectrometric (GC/MS) analysis was carried out with an Agilent 5975 GC-MSD system (Agilent Technologies Inc., Santa Clara, CA). An Innowax FSC column (Hewlett-Packard, HP, USA) (60 m × 0.25 mm, 0.25 μm film thickness) was used with helium as carrier gas (0.8 mL/minute). GC oven temperature was kept at 60 C for 10 minutes and programmed to 220 C at a rate of 4 C/minute, and kept constant at 220 C for 10 minutes and then programmed to 240 C at a rate of 1 C/minute. Split ratio was adjusted 40:1. The injector temperature was at 250 C. MS were taken at 70 eV. Mass range was from 35 to 450 *m/z*.

GC conditions

The GC analysis was carried out using an Agilent 6890N GC system. In order to obtain the same elution order with GC/MS, simultaneous injection was done by using the same column and appropriate operational conditions. The flame ionization detector (FID) temperature was 300 C.

The components of essential oils were identified by comparison of their mass spectra with those in the Baser Library of Essential Oil Constituents, Wiley GC/MS Library, Adams Library, MassFinder Library and confirmed by comparison of their retention indices (RI). Alkanes were used as reference points in the calculation of relative retention indices (RRI). Relative percentage amounts of the separated compounds were calculated from FID chromatograms.

Results and discussion

Mentha pulegium

This species is known locally as filiskin or yarpuz. It use to be cultivated in Turkey for oil and flower water production (15). Previously, all 12 oil samples obtained in 0.3–1.2% yield from Black Sea region were shown to contain 3-oxo compounds. Nine samples had contained pulegone as a main constituent, whereas it did not exist in three samples. The main constituents were as follows: pulegone (25–51%), menthone (5–59%), isomenthone (3–52%) and neomenthol (0.6–19%). Plants collected in İzmir, Balikesir and Bilecik provinces had yielded oils up to 3.3% and seven samples had shown pulegone (53–78%) as the main constituent; a single sample contained isomenthone (68%) and pulegone (15%) (17). In an earlier paper, three different types of oils of Istanbul origin were reported as pulegone/menthone (41–66%/22–44%), menthone/pulegone (43–56%/7–31%) and menthol and its isomers (4–22%) (18). Lawrence reported the main constituents of pennyroyal oil as pulegone, and other 3-oxo-*p*-menthanes and piperitone (26).

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Table 2. *Mentha* species were collected in Turkey.

Mentha pulegium L.
A1: Edirne; Havsa, Environs of Osmanlı Lake, Sedge, 100 m, August 23, 2003, G. Tarımcılar, BULU 30436.
A1: Tekirdağ; Malkara-Tekirdağ, Tekirdağ 27 km, roadside, dry land, 200 m, August 23, 2003, G. Tarımcılar, BULU 30453.
A2: İstanbul; Çatalca, September 2003, H. Malyer, BULU 30463.
A1. Çanakkale; Gelibolu, meadow, 65 m, August 25, 2004, G. Tarımcılar, BULU 30472.
B1: Çanakkale; Bayramiç, Çırpıcılar, 2 km, sandy soil, 400 m, August 26, 2004, G. Tarımcılar, BULU 30484.
B2: Balıkesir; Bandırma, Manyas Bird Sanctuary, göl kenarı, 190 m, August 27, 2004, G. Tarımcılar, BULU 30509.
A2: Bursa; Uluabat Lake, Fadıllı, lakeside, 9 m, September 23, 2004, G. Tarımcılar, BULU 30513.
A2: İstanbul; Şile; Ömerli Dam road, roadside, meadow, August 15, 2005, G. Tarımcılar, BULU 30525.
A2: İstanbul; Çavuşbaşı, Elmalı stream, riverside, meadow, August 16, 2005, G. Tarımcılar, BULU 30533.
A2: Yalova; between Orhangazi-Yalova, Esadiye, 200 m, dry land, September 7, 2006, G. Tarımcılar, BULU 30588.
A2: Yalova; Armutlu-Çınarcık, Çınarcık 13 km, Şenköy, 75 m, September 8, 2006, G. Tarımcılar, BULU 30598.
A2: Yalova; Armutlu-Çınarcık, Çınarcık 10 km, 30 m, dry land, September 8, 2006, G. Tarımcılar, BULU 30599.
A2: Yalova; Termal, road to thermal baths, 100 m, kurak toprak, September 8, 2006, G. Tarımcılar, BULU 30602.
Mentha aquatica L.
A2: İstanbul; Çatalca, September 2003, H. Malyer, BULU 30464.
B2: Balıkesir; exit Balya, Balıkesir-İvrindi junction, Balıkesir 40 km, wet land, 280 m, August 26, 2004, G. Tarımcılar, BULU 30496.
A2: Bursa; Uluabat Lake, Fadıllı, lakeside, 9 m, September 23, 2004, G. Tarımcılar, BULU 30512.
A2: İstanbul; Çavuşbaşı, Elmalı Stream, lakeside, meadow, August 16, 2005, G. Tarımcılar, BULU 30534.
A2: Kocaeli; Maşukiye, Sapanca Lake, 20 m, lakeside, September 7, 2006, G. Tarımcılar, BULU 30585.
M. × *dumetorum* Schultes
A2:Yalova to Kocaeli, Selimiye, 65 m, roadside, dry land, September 7, 2006, G. Tarımcılar, BULU 30582.
Mentha spicata L. ssp. *spicata*
A1: Edirne; Hasköy, riverside, 90 m, August 23, 2003, G. Tarımcılar, BULU 30437.
A1: Tekirdağ-Marmara Ereğlisi, Marmara Ereğlisi 24 km, roadside, 20 m, 24. 08.2003, G. Tarımcılar, BULU 30455.
A1: Tekirdağ-Marmara Ereğlisi, Marmara Ereğlisi 24 km, 24. 08.2003, G. Tarımcılar, BULU 30457.
B2: Balıkesir; Bandırma-Erdek, Tatlısu village centre, roadside, dry land,130 m, August 27, 2004, G. Tarımcılar, BULU 30504.
A2: Yalova; Yalova to İzmit, Sultaniye, 0 m, wet land, September 7, 2006, G. Tarımcılar, BULU 30571,BULU 30572 (two samples).
A2: Yalova road to termal centrum, 140 m, dry land, September 8, 2006, G. Tarımcılar, BULU 30603.
Mentha spicata L. ssp. *tomentosa* (Briq.) Harley
A2: Bursa; İznik-Adapazarı, Kaynarca village, waterfront, 290 m, August 15, 2003, G. Tarımcılar, BULU 30426.
A1: Edirne; Edirne-İstanbul, Oğulpaşa village 15 km, wet land, 70 m, August 23, 2003, G. Tarımcılar, BULU 30434.
A1:Çanakkale; Gelibolu-Keşan, Kalealtı village exit, roadside, 100 m, August 25, 2004, G. Tarımcılar, BULU 30468.
B1: Çanakkale; Bayramiç, Çırpıcılar 2 km, waterfront, Juncus sp.,350 m, August 26, 2004, G. Tarımcılar, BULU 30482.
A2: İstanbul; Ömerli Dam exit, roadside, dry land, 150 m, August 15, 2005, G. Tarımcılar, BULU 30527.
A2: İstanbul; Şile, roadside, meadow, August 15, 2005, G. Tarımcılar, BULU 30530.
A2: İstanbul; Şile, citadel, around Side Restaurant, 30 m, August 15, 2005, G. Tarımcılar, BULU 30531.
A2: Bursa; İnegöl, Oylat, entrance to Gündüzlü village, 420 m, riverside, September 6, 2006, G. Tarımcıla, BULU 30557.
A2: Yalova; Orhangazi to Yalova, Esadiye, 200 m, dry land, September 7, 2006, G. Tarımcılar, BULU 30569.
A2: Yalova; Yalova to İzmit, Sultaniye, 5 m, shade, September 7, 2006, G. Tarımcılar, BULU 30573.
A2: Yalova to Kocaeli, Dereköy, 35 m, dry land, September 7, 2006, G. Tarımcılar, BULU 30574.
A2: Kocaeli; Maşukiye, Sapanca lake, 15 m, wet land, 50 m, September 7, 2006, G. Tarımcılar, BULU 30586.
A2: Yalova; Termal road, Kadıköy 1 km, 25 m, wet land, September 8, 2006, G. Tarımcılar, BULU 30601.
A2: Balıkesir; Marmara Island, Topağacı village, 0 m, roadside, inside water canal, September 10, 2007, G. Tarımcılar, BULU 30627.
M. suaveolens Ehr.
A2: Bursa; İnegöl, Domaniç road, Hilmiye village, 450 m, September 6, 2006, G. Tarımcılar, BULU 30553.
A2: Bursa; İnegöl, Oylat, enterance to Gündüzlü village, 420 m, September 6, 2006, G.Tarımcılar, BULU 30558.
A2: Yalova; Yalova to İzmit, Sultaniye, 0 m, dry land, September 7, 2006, G. Tarımcılar, BULU 30570.
Mentha longifolia (L.) Hudson subsp. *longifolia*
A2: Bursa; İznik-Gemlik, Narlıca, roadside, 210 m, August 15, 2003, G. Tarımcılar, BULU 30430.
A2: Bilecik to Pazaryeri, 9 km, 1000 m, dry land, September 6, 2006, G. Tarımcılar, BULU 30563.
Mentha longifolia (L.) Hudson subsp. *typhoides* (Briq.) Harley var *typhoides*
A2: Bursa; İznik-Adapazarı, Hocaköy cross, roadside, 160 m wet land, August 15, 2003, G. Tarımcılar, BULU 30427.
A2: Bursa; İznik, roadside, 160 m, August 15, 2003, G. Tarımcılar, BULU 30428.
A1: Kırklareli; Kırklareli 22 km, Ürünlü village, waterfront, 120 m, August 23, 2003, G. Tarımcılar, BULU 30439.
A1: Kırkpınar; İnece village, İnece stream bank, 80 m, Juncus sp., August 23, 2003, G. Tarımcılar, BULU 30441.
A1: Kırklareli; Babaeski, Mimar Sinan bridge, river bed, 60 m, August 23, 2003, G. Tarımcılar, BULU 30450.
A1: Çanakkale; Lapseki-Beyçayırı, Lapseki 5 km, wet land, 50 m, August 25, 2004, G. Tarımcılar, BULU 30474.

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Table 2. (*Continued*).

A1: Çanakkale-Çan; Kurşunlu-Dörtyol, waterfront, 55 m, August 25, 2004, G. Tarımcılar, BULU 30477.
B1: Çanakkale-Bayramiç; Bayramiç 11 km, roadside, wet land, 140 m, August 26, 2004, G. Tarımcılar, BULU 30479.
B1: Çanakkale; Bayramiç, Çırpıcılar, dry land, 375 m, August 26, 2004, G. Tarımcılar, BULU 30483.
B1: Çanakkale; Bayramiç to Kazdağı, 10 km, Evciler, waterfront, 315 m, August 26, 2004, G. Tarımcılar, BULU 30485.
B1: Çanakkale; Bayramiç to Kazdağı, 10 km, Evciler, waterfront, 300 m, August 26, 2004, G. Tarımcılar, BULU 30486.
B1: Çanakkale; Bayramiç to Kazdağı, 285 m, August 26, 2004, G. Tarımcılar, BULU 30487.
B1: Çanakkale; Bayramiç, Kazdağı, wet land, 450 m, August 26, 2004, G. Tarımcılar, BULU 30488.
B1: Çanakkale; Karaköy-Çan, Kızılelma village, waterfront, 950 m, August 26, 2004, G. Tarımcılar, BULU 30490.
B1: Çanakkale; Karaköy-Çan, Kızılelma village, waterfront, 950 m, August 26, 2004, G. Tarımcılar, BULU 30491.
B2: Çanakkale; Yenice, waterfront, 350 m, August 26, 2004, G.Tarımcılar, BULU 30492.
B2: Balıkesir; Balya exit, Balıkesir-İvrindi yol ayrımı, Balıkesir 40 km, wet land, 280 m, August 26, 2004, G. Tarımcılar, BULU 30495.
B2: Balıkesir; Konakpınar road, Taşköy, waterfront, 300 m, August 26, 2004, G. Tarımcılar, BULU 30497.
B2: Balıkesir-Kütahya; Kepsut, riverside, 180 m, August 27, 2004, G. Tarımcılar, BULU 30500.
B2: Balıkesir-Kütahya; Kepsut exit, roadside, 200 m, August 27, 2004, G. Tarımcılar, BULU 30501.
B2: Balıkesir; Bandırma-Erdek, Tatlısu village centre, roadside, dry land,130 m, August 27, 2004, G. Tarımcılar, BULU 30505.
A2: Bursa; Uluabat gölü, entrance to Akçapınar village, waterfront, 52 m, September 23, 2004, G. Tarımcılar, BULU 30515.
A2: Bursa; İnegöl, Oylat, 750 m, September 6, 2006, G.Tarımcılar, BULU 30554.
A2: Bursa; İnegöl, Oylat to Hilmiye, 700 m, roadside, wet land, September 6, 2006, G. Tarımcılar, BULU 30555.
A2: Bursa; İnegöl, Oylat to Hilmiye, 3km, 700 m, September 6, 2006, G. Tarımcılar, BULU 30556.
A2: Bursa; İnegöl, Oylat, entrance to Gündüzlü village, 420 m, riverside, September 6, 2006, G. Tarımcılar, BULU 30559.
A2: Bursa; entrance to İnegöl-Pazaryeri road, Hasanpaşa, 350 m, roadside, September 6, 2006, G. Tarımcılar, BULU 30560.
A2: Bursa; Yenişehir 1 km. İncirli village, 300 m, waterfront, September 6, 2006, G. Tarımcılar, BULU 30565.
A2:Yalova to Kocaeli, Selimiye, 75 m, dry land, September 7, 2006, G. Tarımcılar, BULU 30579.
A2: Kocaeli; Yazlık township, 5 m, dry land, September 7, 2006, G. Tarımcılar, BULU 30583.
A2: Kocaeli; Maşukiye villas before function, 10 m, waterfront, September 7, 2006, G. Tarımcılar, BULU 30590.
A2: Bursa; Gemlik, Küçükkumla village centre, 20 m, dry land, September 8, 2006, G. Tarımcılar, BULU 30591.
A2: Bursa; Gemlik, entrance to BüyükKumla village, 5 m, dry land, September 8, 2006, G. Tarımcılar, BULU 30593.
A2: Bursa; Gemlik, Narlı, 3 m, wet land, fountain side, September 8, 2006, G. Tarımcılar, BULU 30594.
A2: Yalova; Armutlu to Çınarcık, 25 km, 150 m, waterfront, September 8, 2006, G. Tarımcılar, BULU 30595.
A2: Yalova; Armutlu to Çınarcık, 22 km, Esenköy, 0 m, waterfront, September 8, 2006, G. Tarımcılar, BULU 30596.
A2: Yalova; Armutlu to Çınarcık, 13 km, Şenköy, 75 m, September 8, 2006, G. Tarımcılar, BULU 30597.
A1: Balıkesir; Marmara island, entrance to Topağacı village, 0 m, meadow, September 10, 2007, G. Tarımcılar, BULU 30623.

Mentha × *rotundifolia* (L.) Hudson

B2: Balıkesir; Bandırma-Erdek, Düzler exit, meadow, 130 m, August 27, 2004, G. Tarımcılar, BULU 30508.

Mentha × *villosa-nervata* Opiz

A1: Edirne; Edirne-İstanbul, Oğulpaşa village15 km, wet land, 70 m, August 23, 2003, G. Tarımcılar, BULU 30434.
A1: Çanakkale; Saros körfezi, Kocaçeşme village, dry river bed, 35 m, August 25, 2004, G. Tarımcılar, BULU 30470.
B2: Balıkesir; Bandırma-Erdek, Tatlısu village centre, roadside, dry land,130 m, August 27, 2004, G. Tarımcılar, BULU 30506.
A2: Bursa; İnegöl, İnegöl to Oylat, garden side, wet land, July 17, 2005, G. Tarımcılar, BULU 30523.
A2: İstanbul; Ömerli Dam, around public dwelling, wet land, August 15, 2005, G. Tarımcılar, BULU 30526.
A2: Yalova to Kocaeli, Selimiye, 65 m, waterfront, September 7, 2006, G.Tarımcılar, BULU 30580.

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In the present work, 13 samples were studied and the oil yields ranged between 0.9% and 2.4%. The main constituents were found to be isomenthone (23–74%), pulegone (trace–57%), menthone (1–34%) and piperitone (up to 18%). Eleven oils contained isomenthone/pulegone (29–74%/10–57%); one oil contained pulegone/menthone (36%/34%) and another oil contained piperitone (95%) as the main constituents. Compared with the oils of the Black sea region, in the oils of the Marmara region isomenthone seemed to predominate over menthone.

Mentha aquatica

Previously, 16 oil samples from Black sea region obtained in 0.1–0.5% yield were analyzed. Eleven oils contained menthofuran (21–48%) as the main constituent and sesquiterpene-rich oils containing elemol, β-caryophyllene and viridiflorol were also encountered (17, 28). In another study with Turkish samples, menthofuran (35%) was found as the main constituent and another sample was reported to contain menthol (37%), menthyl acetate (33%) and menthofuran (17%) as the main constituents (19). According to Lawrence, menthofuran appears to be the main constituent of *M. aquatica* oils despite a few exceptions (26).

In the present study, oil yield was found to be 0.4–0.5% in five samples. The main constituents included menthofuran (14–58%), 1,8-cineole (5–27%), β-caryophyllene (7–16%), viridiflorol (3–11%), limonene (3–7%) and β-pinene (1–5%). Two samples contained menthofuran (35% and 58%) as the main

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constituent, whereas the remaining three samples contained menthofuran/1,8-cineole (14–30%/15–27%) as major compounds. The main sesquiterpenes included β-caryophyllene and viridiflorol as reported previously; however, no elemol was detected.

Mentha × *dumetorum*

This plant is a hybrid of *M. aquatica* and *M. longifolia*. Previously, oil yields in seven samples of Black sea origin were reported to be 0.2–1.5%. Four samples had menthofuran/1,8-cineole (18–41%/8–13%) as the main constituents, whereas one of them contained menthyl acetate (11%), pulegone (9%), menthol (7%) and menthone (7%) (17).

Essential oil obtained in 0.4% from a single sample contained menthofuran (28%), menthyl acetate (18%), β-caryophyllene (12%), menthol (9%), germacrene D (6%), pulegone (3%) and menthone (3%).

Previous studies reported by others showed similar results. In those studies, samples containing menthofuran/pulegone/menthol (25%/17%/11%) and menthyl acetate/menthofuran/pulegone (33%/27%/10%) were encountered as the main constituents (9, 26).

Mentha spicata subsp. *spicata*

This species is known as bahçe nanesi (garden mint), kıvırcık nane (curly mint) or Antep nanesi (Antep mint) in Turkey. It is cultivated in home gardens throughout Turkey and commercially in Gaziantep. Dried leaves of this species are the most widely consumed mint in Turkey. It is known as spearmint in world markets and its l-carvone-rich cultivar is widely cultivated. Oil production of this species in USA is 500 tons annually (27).

In our previous study on three samples collected in Kastamonu province in northern Turkey, oil yields were rather low (0.6–0.8%) and carvone contents were also low (0%, 14% and 19%) (17).

In the seven samples studied, the oil yields were found to be 1.2–2.7%. Carvone (59–77%), limonene (2–23%), 1,8-cineole (1–7%) and *trans*-dihydrocarvone (1–4%) were the main constituents. In one sample collected from Hasköy (Edirne), the main constituents were characterized as isomenthone (32%), *cis*-piperitone oxide (30%) and menthone (14%).

Mentha spicata subsp. *tomentosa*

Oil yields were 0.2–2.6% in 14 samples. The main constituents were as follows: piperitenone oxide (1–74%), *trans*-piperitone oxide (0–61%), *cis*-piperitone oxide (0–11%), 1,8-cineole (1–9%) and germacrene D (0–7%). In a single oil sample, the main constituents were found to be isopulegyl acetate (30%), isopulegol (21%) and pulegone (15%).

No previous study has been encountered on the oil of this taxon. In a recent revision, this taxon was given as a synonym of *Mentha spicata* subsp. *condensata* (Briq.) Greuter et Burdet (6). Two oil analyses were encountered under this name giving carvone (57%) and piperitenone oxide (68%), respectively (26).

Mentha suaveolens

Several studies exist on the essential oil of this species locally known as 'Anuk' in Turkey. However, there is only one previous report on the oil sample from a material collected in Sakarya reporting 0.7% oil yield and piperitenone oxide (47%) and *trans*-piperitone oxide (26%) as the main constituents (17). Other studies on oils of other origins reported piperitenone oxide as the main constituents. The other major constituents included 1,2-epoxy-3-oxo-compounds such as *cis*- and *trans*-piperitone oxides, 2-oxo-compounds like carvone and a reduced 2-oxo-compound like dihydrocarvone (26).

In the present study, three samples with 0.3–1.0% oil yields contained the following main constituents: piperitenone oxide (63–77%), limonene (2–6%), 1-octen-3-yl acetate (1–5%), β-pinene (2–3%), myrcene (2–3%) and α-pinene (2–3%).

Mentha longifolia subsp. *longifolia*

Mentha longifolia is known as anik, kinzi, it nanesi (dog mint) or tüylü nane (hairy mint) in Turkey (28). In our previous study, oil yield on 18 species collected from the Black sea region were found to be 0.1–1.0% and *cis*- and *trans*–piperitone oxides were the main constituents in seven samples. Four samples had contained linalool and three samples had contained carvone. One sample each had contained isomenthone, α-terpinyl acetate and terpinen-4-ol as the main constituents, respectively (17). Lawrence reported piperitone oxide, carvone and linalool chemotypes. One of the study materials was obtained from Izmir, Turkey, which had contained carvone (68%) and *trans*-carvyl acetate (21%) as the main constituents (7). Another previous study with Turkish material had reported menthyl acetate (0–47%), menthone (6–46%), menthol (16–30%) and menthofuran (0–7%) as the main constituents (19).

In Greece, piperitone oxide-rich chemotypes and in Italy, piperitenone oxide-rich chemotypes were reported (8, 9, 29). Kokkini reported the occurrence of carvone chemotype in oil-rich wild *M. longifolia* populations in Crete (30). In the oils of two Israeli samples 1,8-cineole (29%), piperitone (14%) and *cis*-piperitone oxide

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(15%) was found as the main constituent in one, and pulegone (73%) in the other (31). Lawrence suggested a thorough investigation of the chemical diversity of the essential oils of this taxon (8, 9, 25).

In the present study, two samples gave 0.4% and 1% oil yields. The main constituents of the oil rich sample were carvone (52%), limonene (14%) and 1,8-cineole (13%). However, an unknown compound with RI: 2209 at GC (35%) (Figure 1) and 1,8-cineole (15%) were found as the main constituents.

Mentha longifolia subsp. *typhoides* var. *typhoides*

Previously, our group had reported on the oil compositions of 19 samples collected from northern Turkey in which *trans*-piperitone oxide (59–72%) in five samples; *cis*-piperitone oxide (61–62%) in two samples; linalool (64–87%) in three samples and linalool/1,8-cineole (27%/18%) in one sample; carvone (39–59%) in two samples; *trans*-sabinene hydrate (51–71%) in two samples; *trans*-sabinene hydrate (26%) and carvone (25%) in one sample; and menthone/*trans*-piperitone oxide (31%/24%), β-caryophyllene/*trans*-piperitone oxide/caryophyllene oxide (12%/13%/11%), caryophyllene oxide/*trans*-piperitone oxide/β-caryophyllene (29%/13%/12%) in one sample each were main constituents (17).

Oils from samples collected in South Anatolia indicated piperitone (63%), pulegone (42%), 1,8-cineole (58%) and 1,8-cineole/pulegone (31%/85%) chemotypes (32). Lawrence had reported on piperitenone and *trans*-piperitone oxide chemotypes (26).

In the present study, oil yields ranged between 0.3% and 2.0% in 38 samples. Three characteristic compounds in 29 oil samples were piperitenone oxide (31–85% in 11 samples), *trans*-piperitone oxide (33–80% in 16 samples) and *cis*-piperitone oxide (24–33% in two samples). The main constituents in the remaining 12 samples were isopulegol (25–80% in 3 samples), iso-isopulegol (22%), iso(iso)pulegyl acetate (17%), pulegone (32%), carvone (73%) and piperitenone (65%) together with an unknown compound at RI: 2209 at GC in four samples (6–35%) (Figure1). Furthermore, in two samples, this unknown compound was found as the second major compound (6–24%).

Mentha × *rotundifolia*

There is no previous study on the oil of this hybrid from Turkey. According to Lawrence, *cis*-dihydrocarvone (69%), carvone (60%), menthyl acetate (51%), piperitone oxide (75%) and piperitenone oxide (74–77%) have been reported (26). Linalool (95%), carvone (52%) and menthol (88%)-rich mutants were produced in Japan by X- and gamma-ray radiations (33).

In the present work, piperitenone oxide (64%), α-pinene (4%), myrcene (3%), germacrene D (2%) and sabinene (2%) were found as the main constituents in a single oil sample obtained in 0.6% yield.

Mentha × *villoso-nervata*

This species was recorded in the Flora of Turkey recently (34, 35). In our previous study, two samples from northern Turkey, which yielded 0.4% and 0.6% oil, were shown to belong to two chemotypes: piperitone oxide/dihydrocarvone (40%/27%) and dihydrocarvone/carvone (58%/10%) (17). The existing two chemotypes – piperitenone oxide/piperitone oxide (69%/66%) and carvone/dihydrocarvone (43%/16%) – was reported from Greece (36). The occurrence of a carvone-rich (70–80%) type was reported from Crete

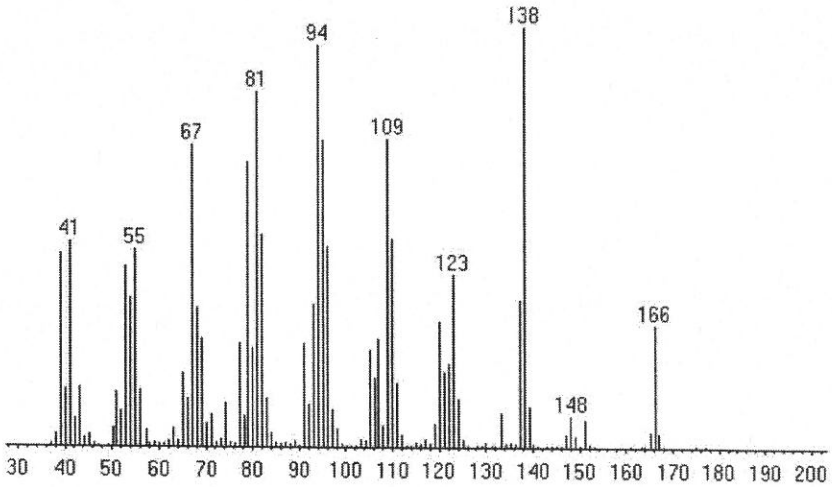

Figure 1. Mass spectrum of unknown compound (RI: 2209) in *Mentha longifolia* subsp. *longifolia* essential oil.

Table 3. Summary of the cumulative analytical results on the oils of *Mentha* taxa from the Marmara Region of Turkey.

Section – group	*Mentha*	No.	1-oxy	2-oxo	3-oxo	3-oxo-1,2-epoxy	1,8-epoxy
Pulegium	*pulegium*	13			13[c,d,e,g]		
Mentha – Spicatae	*spicata/spicata*	7		6[a]	1[d]		
Mentha – Spicatae	*spicata/tomentosa*	14			1[i]	13[j,k]	
Mentha – Spicatae	*suaveolens*	3				3[i]	
Mentha – Spicatae	*longifolia/longifolia*	2*		1[a]			
Mentha – Spicatae	*longifolia/typhoides/typhoides*	38*		1[a]	4[h,i,e]	29[j,k]	
Mentha – Spicatae	× *villoso -nervata*	6	1		1[e]	4[k]	
Mentha – Spicatae	× *rotundifolia*	1				1[j]	
Mentha – Capitatae	× *dumetorum*	1			1[f]		
Mentha – Capitatae	*aquatica*	5			3[f]		2[m]
*Unknown (5)							
	Total	90	1	8	24	50	2

		Number	Percentage
1-oxygenated	*trans*-sabinene hydrate	1	1
2-oxo	[a]carvone,[b]dihydrocarvone (*cis/trans*)	8	9
3-oxo	[c]menthone, [d]isomenthone, [e]pulegone, [f]menthofuran, [g]piperitone, [h]piperitenone, [i]isopulegol	24	27
3-oxo-1,2-epoxy	[j]piperitenone oxide, [k]piperitone oxide (*cis/trans*)	50	56
1,8-epoxy	[m]1,8-cineole	2	2
Unknown		5	5
		90	100

Notes: [a]carvone, [b]dihydrocarvone (*cis/trans*), [c]menthone, [d]isomenthone, [e]pulegone, [f]menthofuran, [g]piperitone, [h]piperitenone, [i]isopulegol, [j]piperitenone oxide, [k]piperitone oxide (*cis/trans*), [m]1,8-cineole.

and it was speculated to be the remnant of an ancient cultivar (30).

In the course of the present work, six samples were studied. Oil yields ranged between 1.0% and 2.2%. Four samples gave the following main constituents: *trans*-piperitone oxide (58–76%), piperitenone oxide (2–13%), 1,8-cineole (1–7%), *cis*-piperitone oxide (2–5%), β-caryophyllene (2–5%), germacrene D (0–4%) and limonene (1–3%). The main constituents of a single sample were *trans*-sabinene hydrate (42%) and pulegone (16%).

Conclusions

Kokkini classified mint oils according to chemical types as acyclic, 2-substituted and 3-substituted based on previous results in her two important reviews (30). We followed the same in our previous study on the mint oils of northern Turkey (17).

We reported the occurrence of 3-oxo compounds in 67% of the North Anatolian Mint samples in which 1,2-epoxy compounds were main constituents. In the present study, 3-oxo compounds constituted 81% of the samples. 3-Oxo compounds like menthone, isomenthone, menthofuran, pulegone, isopulegol, iso(iso)pulegol, isopulegyl acetate, piperitone and piperitenone constituted 25% of the oils, whereas 3-oxo-1,2-epoxy compounds such as piperitenone oxide and *cis*-/*trans*-piperitone oxides constituted 56% of all the oils studied (Table 3).

We intend to continue studying the mint oils of other regions in Turkey in the same fashion.

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