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Composition and Antimicrobial Activities of Volatile Components of *Minuartia meyeri*

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The essential oil of air-dried *Minuartia meyeri* (Boiss.) Bornm. (Caryophyllaceae) was obtained by hydrodistillation in a Clevenger-type apparatus and analyzed by GC-MS. Fifty-two components were identified in the oil. The main components in the essential oil of *M. meyeri* were nonacosane (6.2%), 6,10,14-trimethyl-2-pentadecanone (5.1%), nonanal (4.6%), and β-caryophyllene (2.9%). The antimicrobial activity of the isolated essential oil of the plant was also investigated and it showed moderate antibacterial activity against Gram-positive and Gram-negative bacteria, but no antifungal activity was observed against 2 yeast-like fungi.

Key Words: *Minuartia meyeri*, Caryophyllaceae, Essential oil, Antimicrobial activity, GC-MS.

Introduction

The genus *Minuartia* L. (Caryophyllaceae) comprises 100 species that are distributed over a large part of Central and Southern Europe, and West, Central, and Southern Asia.[1] The genus *Minuartia* is represented in Turkey by 50 taxa including 17 varieties and 21 subspecies. One of the non-endemic plants of the genus is *Minuartia meyeri*, which is distributed mainly in Central and Northeast Anatolia[1−3].

Essential oils are natural mixtures of terpenes/terpenoids, most of which are obtained from aromatic and medicinal plants. The chemical composition of essential oil differs in each species or subspecies and is characteristic for the species in question[4−7]. Identification of individual components of complex mixtures such as terpenes/terpenoids in essential oils requires the use of several techniques. One of the most popular methods of studying essential oil composition is gas chromatography-mass spectrometry (GC-MS), which

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allows the identification of the specific natural compounds found in an essential oil by comparing their relative retention times/indices and their mass spectra[4−14].

To our knowledge, there are no published reports on the chemical composition and antimicrobial activity of the essential oil of *M. meyeri*. As part of this systematic research, the essential oil of the plant was obtained by hydrodistillation in a Clevenger-type apparatus[4,5]. The crude essential oil was then investigated by GC-MS[4−14]. Fifty-two components of various types were identified from the sample. The compounds were identified by a typical library search (NIST, WILEY). Therefore, we focused on the chemical composition and antimicrobial property of the essential oil of *M. meyeri*[15,16].

Experimental

Plant material

M. meyeri (Boiss.) Bornm. (Caryophyllaceae) was collected in Sümela Maçka-Trabzon (A7) (open and stony place at a height of ∼1900 m) in northeastern Turkey in July 2004. A voucher specimen (No. Coşkunçelebi 495-2004, KTUB) was deposited in the herbarium of the Department of Biology, Karadeniz Technical University, Turkey. The plant was identified[1−3] immediately after collection and air-dried at room temperature for later analysis.

Isolation of the essential oil

The air-dried plant (35 g) of *M. meyeri* was hydrodistilled in a Clevenger-type apparatus using an ice bath as cooling system (3 h). The oil was taken by dissolving in HPLC grade *n*-hexane (0.5 mL) and kept at 4 ^{o}C in a sealed brown vial. Then 1 μL of the extract was directly injected into the GC-MS instrument. The percentage yield of the oil was calculated on a moisture-free basis (0.20 $\pm$ 0.1, v/w).

Gas chromatography

GC-MS analysis was performed using an Agilent-5973 Network System. A mass spectrometer with an ion trap detector in full scan under electron impact ionization (70 eV) was used. The chromatographic column for the analysis was an HP-5 capillary column (30 m x 0.32 mm i.d., film thickness 0.25 μm). The carrier gas used was helium at a flow rate of 1 mL/min. The injection was performed in splitless mode at 230 °C. Essential oil solution (1 μL) in hexane (HPLC grade) was injected and analyzed with the column held initially at 60 °C for 2 min and then increased to 260 °C with a 5 °C/min heating ramp and subsequently kept at 260 °C for 13 min. The relative percentage amounts of the separated compounds were calculated from total ion chromatograms by a computerized integrator.

Antimicrobial activity assessment

All test microorganisms were obtained from the Refik Saydam Hifzissihha Institute (Ankara, Turkey) and were as follows: *Escherichia coli*, *Klebsiella pneumoniae*, *Yersinia pseudotuberculosis*, *Serratia marcescens*, *Enterococcus faecalis*, *Staphylococcus aureus*, *Bacillus subtilis*, *Candida albicans*, and *Candida tropicalis*. Essential oil was dissolved in acetone to prepare extract stock solution of 500 μg/mL.

Agar-well diffusion method

A simple susceptibility screening test using an agar-well diffusion method[15] as adapted earlier[16] was used. Each microorganism was suspended in Brain Heart Infusion (BHI) (Difco, Detroit, MI) broth and diluted approximately 10^6 colony forming unit (cfu) per mL. They were "flood-inoculated" onto the surface of BHI agar and Sabouraud Dextrose Agar (SDA) (Difco, Detriot, MI) and then dried. For *C. albicans* and *C. tropicalis*, SDA was used. Five-millimeter diameter wells were cut from the agar using a sterile cork-borer, and 100 μL of the extract solutions were delivered into the wells. The plates were incubated for 18 h at 35 °C. Antimicrobial activity was evaluated by measuring the zone of inhibition against the test microorganism. Ceftazidime (Fortum) (10 μg) and Triflucan (5 μg) were the standard drugs for antibacterial and antifungal activities, respectively. Acetone was used as solvent control. The tests were carried out in duplicate. The results were interpreted in terms of diameter of inhibition zone: (-): $<$ 5.5 mm; (+): 5.5-10 mm; (++): 11-15 mm; (+++): $\geq$ 16 mm. The results are shown in Table 3.

Results and Discussion

The essential oil, which was pale yellow, was obtained by hydrodistillation in a Clevenger-type apparatus[4–6] from *M. meyeri* with the yield of 0.20 $\pm$ 0.1 (v/w) on a dry-weight basis. The general chemical profile of the essential oil, the percentage content, and retention indices of the constituents are summarized in Table 1. The essential oil of *M. meyeri* was analyzed by GC-MS with an HP-5 column, and 52 components were identified on the basis of a typical library search, selecting only the components showing matches exceeding 80%, which represented about 68.2% of the total detected constituents[4–14]. Nonacosane (6.2%), 6,10,14-trimethyl-2-pentadecanone (5.1%), nonanal (4.6%), and β-caryophyllene (2.9%) were the major compounds in the essential oil.

Table 1. Identified components in the essential oil of *Minuartia meyeri* (Boiss.) Bornm.[a,b].

No.	Compound	Area %	Exp. RI	Identif. (LRI/MS)	Ref.
1	Octanal	0.2	998	1001	c
2	1-Octanol	0.4	1068	1070	c
3	Nonanal	4.6	1105	1102	c
4	1-Nonanol	0.3	1174	1171	c
5	Decanal	0.8	1205	1204	c
6	β-Cyclocitral	0.6	1221	1218	d
7	(*E*)-2-Decenal	0.8	1263	1261	c
8	Theaspirane A	0.4	1298	1300	e
9	Decyloxirane	0.3	1307	MS	f
10	(*E*,*E*)-2,4-Decadienal	1.2	1316	1314	c
11	1,2-Dihydro-1,5,8-trimethylnaphthalene	0.5	1354	MS	f
12	Farnesane	0.8	1378	MS	f
13	β-(*E*)−Damascenone	0.7	1385	1380	c
14	1,2-Dimethyl-3-ethenyl-1,4-cyclohexadiene	0.6	1394	MS	f
15	Tetradecane	0.7	1400	MS	f
16	β-(*E*)-Caryophyllene	2.9	1421	1418	c

Table 1. Contunied.

No.	Compound	Area %	Exp. RI	Identif. (LRI/MS)	Ref.
17	Neryl acetone	0.9	1435	1434	c
18	Trans-β-Farnesene	0.8	1459	1458	c
19	2,6,10,14-Tetramethylheptadecane	1.2	1464	MS	f
20	Germacrene D	0.9	1483	1480	c
21	β-Ionone	1.0	1487	1485	c
22	Pentadecane	1.7	1500	MS	f
23	2,5-Bis(1,1-dimethylethyl)phenol	1.4	1514	MS	f
24	Farnesal	0.7	1543	MS	f
25	Dodecanoic acid	2.8	1569	1568	c
26	Caryophyllene oxide	2.3	1584	1581	c
27	Hexadecane	2.1	1600	1600	g
28	1-Heptadecene	3.6	1689	1692	d
29	Heptadecane	1.5	1700	1700	g
30	(*E*,*E*)-Farnesol	4.3	1724	1722	h
31	Tetradecanoic acid	1.4	1764	1769	h
32	Hexadecyl oxirane	0.9	1779	MS	f
33	Octadecane	0.5	1800	1800	f
34	6,10,14-Trimethyl-2-pentadecanone	5.1	1847	1846	i
35	Nonadecane	0.6	1900	1900	g
36	Oxacycloheptadecan-2-one	0.4	1932	MS	f
37	2,8-Dimethyldibenzothiophene	0.6	1947	MS	f
38	3,4,5-Trimethoxy benzoic acid	0.7	1969	MS	f
39	4,9-Dimethylnaphtho[2,3-b]thiophene	1.3	1991	MS	f
40	Eicosane	0.6	2000	2000	g
41	1-Hydroxy-9H-xanthen-9-one	0.8	2006	MS	f
42	Cyclotetradecane	0.9	2084	MS	f
43	*cis*-Phytol	2.3	2113	2114	g
44	Docosane	0.4	2200	2200	g
45	Tricosane	0.5	2300	2300	g
46	Tetracosane	0.5	2400	2400	g
47	1-Eicosanal	0.4	2491	MS	f
48	Pentacosane	0.7	2500	2500	g
49	Hexacosane	0.7	2600	2600	g
50	Heptacosane	1.2	2700	MS	f
51	Cyclooctacosane	0.5	2795	MS	f
52	Nonacosane	6.2	2900	MS	f
	Total isolate	68.2			
	Unknown	3.0			

[a] Compounds are listed in order of elution.

[b] RI (retention index) calculated from retention times relative to that of *n*-alkanes (C_6-C_{32}) on the non-polar HP-5 column.

[c] Adams (1995).

[d] Bertoli et al. (2004).

[e] Leffingwell et al. (2005).

[f] NIST and Wiley Libraries.

[g] Skaltsa et al. (2003).

[h] Oyedeji et al. (2005).

[i] Yaylı et al. (2005).

The chemical class distribution and major components of the essential oil of the plant are reported in Table 2. The compounds were separated into 10 classes, namely monoterpen, monoterpenoids, sesquiterpenes, sesquiterpenoids, diterpenoid, aldehydes, benzene compounds, heterocyclics, hydrocarbones and others (Table 2). Hydrocarbon components were the major constituents of essential oil, at a rate of 24.7%.

Table 2. The chemical class distribution and the main components in each class of the essential oil of *Minuartia meyeri*.

Compounds class	Area %	Number of comp.	Major component	RI	Area %
Monoterpen	0.8	1	Farnesane	1378	0.8
Monoterpenoids	2.5	3	β-Ionone	1487	1.0
Sesquiterpenes	1.7	2	Germacrene D	1483	0.9
Sesquiterpenoids	10.2	4	(*E*,*E*)-Farnesol	1724	4.3
Diterpenoid	2.3	1	*cis*-Phytol	2113	2.3
Aldehydes	8.0	6	Nonanal	1105	4.6
Benzene compounds	2.1	2	2,5-Bis(1,1-dimethyl-ethyl)phenol	1514	1.4
Heterocyclics	4.3	6	4,9-Dimethylnaphtho [2,3-b]thiophene	1991	1.3
Hydrocarbons	24.7	19	Nonacosane	2900	6.2
Others	11.6	8	6,10,14-Trimethyl-2-pentadecanone	1847	5.1
Unknown	3.0	4			
Total	71.2	56			

Table 3. Screening results for antimicrobial activity of the essential oil components from *Minuartia meyeri*.

Sample	Stock (μg/mL)	Microorganisms and inhibition zone (mm)								
		Ec	Yp	Kp	Sm	Ef	Sa	Bs	Ca	Ct
M. meyeri	500	-	+	-	-	+	+	-	-	-
Ceftazidime	10	+++	+++	+++	+++	+++	+++	+++		
Triflucan	5								+++	+++

Results were interpreted in terms of the diameter of the inhibition zone: (-): $<$ 5.5 mm; (+): 5.5-10 mm; (++): 11-15 mm; (+++): $\geq$ 16 mm.
Ec: *Escherichia coli* ATCC 35218, Yp: *Yersinia pseudotuberculosis* ATCC 911, Kp: *Klebsiella pneumoniae* ATCC 13883, Sm: *Serratia marcescens* ATCC 13880, Ef: *Enterococcus faecalis* ATCC 29212, Sa: *Staphylococcus aureus* ATCC 25923, Bs: *Bacillus subtilis* ATCC 6633, Ca: *Candida albicans* ATCC 60193, Ct: *Candida tropicalis* ATCC 13803.

The antimicrobial activity for the essential oil of *M. meyeri* was tested in vitro using the agar-well diffusion method with the microorganisms as seen in Table 3. The essential oil showed antibacterial activity against Gram-positive and Gram-negative bacteria, but no antifungal activity was observed against the 2 yeast-like fungi[15,16].

The test extracts showed better antimicrobial activity against Gram-positive bacteria than against Gram-negative bacteria. The essential oil extract of *M. meyeri* showed antibacterial activity against *Y. pseudotuberculosis*, *E. faecalis* and *S. aureus*, but no antimicrobial activity was observed against the bacteria *E. coli*, *K. pneumoniae*, *S. marcescens* and *B. subtilis*, and the fungi *C. albicans* and *C. tropicalis*. This is the first chemical composition analysis performed by a GC-MS analytical method, and antimicrobial activity report for the essential oil of *M. meyeri*.

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