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RESEARCH REPORT

Chemical Composition of Myrtle Leaf Essential Oil from Corsica (France)

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Abstract

Collective and individual samples of myrtle leaf oil from Corsica were analyzed by GC, GC/MS and Carbon-13 NMR spectroscopy. Corsican myrtle was found to contain α-pinene (47.9-59.5%) and 1,8-cineole (19.8-28.1%) as major components. It belongs to the α-pinene/1,8-cineole chemotype.

Key Word Index

Myrtus communis subsp. *communis*, Myrtaceae, myrtle, essential oil composition, α-pinene, 1,8-cineole, chemotype.

Introduction

Myrtle (*Myrtus communis* L.) is an evergreen shrub belonging to the family of Myrtaceae. It grows spontaneously throughout the Mediterranean area (1,2). The essential oil, obtained by steam distillation of twigs, leaves and sometimes flowers and berries is used both in flavor and fragrance compositions. Myrtle berries are also employed in the formulation of bitters and liquors.

The chemical composition of myrtle oils from various origins has been previously reported in the literature (3,4). Most of the oils have a high content of 1,8-cineole (19-45%). Among them, two groups can be distinguished according to the presence in noticeable amounts or the lack of myrtenyl acetate. Each group can further be divided into two subgroups in accordance with the relative ratio of α-pinene and myrtenyl acetate, or α-pinene and 1,8-cineole.

In the first group, Spanish oil was characterized by a high content of myrtenyl acetate (35.9%) and a low content of α-pinene (8.2%) (5,6), while oils from Morocco (5,7,8), Portugal (5,9), France (Esterel) (8), Albania (5) and Yugoslavia (7) showed appreciable amounts of α-pinene (18.5-24.8%) and myrtenyl acetate (9.0-25.0%).

In the second group, oils from Lebanon (7) and Iran (10) contained similar amounts of α-pinene and 1,8-cineole, while those from Tunisia (7) and Sardinia (11) differed from others by a high content of α-pinene (51-53%) which is more than twice as high as that of 1,8-cineole.

In contrast, oils which were characterized by an appreciable content of linalool and limonene in addition to α-pinene and 1,8-cineole, have been found in Greece (8), California (3a) and Russia (3b).

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Table I. Percentage composition of Corsican commercial oils of *Myrtus communis*

Components	A	B	C	D	E	F	G	H	Min.	Max.	Aver.
α-pinene	47.9	50.3	53.8	54.3	54.9	55.9	58.1	59.5	47.9	59.5	54.3
isobutyl isobutyrate	0.8	0.8	0.6	0.6	0.6	0.7	0.9	0.4	0.4	0.9	0.7
β-pinene	0.6	0.6	0.7	0.6	0.7	0.5	0.6	0.6	0.5	0.7	0.6
δ-3-carene	0.4	0.4	0.7	0.3	0.7	0.4	0.5	0.6	0.3	0.7	0.5
myrcene	0.8	0.8			0.4					0.8	
α-phellandrene					0.8					0.8	
isobutyl 2-methylbutyrate	0.5	0.4	0.3	0.3	0.4		0.6	0.5		0.6	0.4
limonene	5.9	5.9	4.7	5.8	4.6	4.7	5.9	5.3	4.6	5.9	5.3
1,8-cineole	27.3	28.1	25.9	24.5	25.0	25.7	19.8	21.1	19.8	28.1	24.7
γ-terpinene	0.8	0.7	0.6	0.3	0.8		0.7	0.5		0.8	0.6
(E)-β-ocimene			0.8		0.9		0.4	0.4		0.9	
p-cymene	1.4	1.6	1.3	0.8	0.9	1.3	0.6	1.4	0.6	1.6	1.2
terpinolene	0.7	0.7	1.1	0.5	1.3		1.3	1.0		1.3	0.9
linalool	1.6	1.3	2.2	1.6	2.0	1.6	1.8	2.7	1.3	2.7	1.8
terpinen-4-ol	1.2	0.7									
α-terpineol	1.3	1.3	1.4	1.3	1.3	1.4	1.5	1.3	1.3	1.5	1.3
geranyl acetate	1.3	1.1	1.7	1.8	1.5	1.8	1.7	1.0	1.0	1.8	1.5
methyl eugenol	0.5	0.5	0.4	0.7	0.4	0.6	0.4	0.6	0.4	0.7	0.5
Total	93.0	95.2	96.2	93.4	97.2	94.6	94.8	96.9	93.0	97.2	

the reported percentages are obtained by GC on the polar column

Oils rich in 1,8-cineole, terpinen-4-ol and α-pinene have been found in France (12) and Morocco (12). Finally, oils obtained by hydrodistillation of myrtle berries (ripe or unripe) exhibited chemical compositions quantitatively different from those of the leaves (6,13).

Little is known about Corsican myrtle leaf oil. Only one study described the chemical composition of two commercial samples (7). Consequently, as part of our on-going work on the chemical composition of oils obtained from wild growing plants of Corsica (14-16), we investigated several samples of *M. communis*.

The oils were analyzed by GC, GC/MS, and Carbon-13 NMR. This latter technique follows a methodology developed in our laboratory (17,18) and appears to be particularly well-suited for chemical polymorphism studies (14-16,19).

Experimental

Sampling: Plant material of *M. communis* subsp. *communis* was collected in different parts of Corsica. The twenty-four individual samples including M1, M2 and M3, were harvested during May 1994 on the coastline (elevation 0-200 m), from shrubs of about the same state of maturity (young branches).

Essential Oil: The oils were obtained by hydrodistillation of fresh aerial parts with industrial apparatus (collective oil) or a Clevenger-type apparatus for 3 h (individual oils; yields: 0.13-0.46%).

Components Identification: Identification of known compounds is based on comparison of their MS and/or ^{13}C-NMR spectra with those of authentic samples together with the retention indices (against a homologous series of n-alkanes) on two columns of different polarity. Peak area percentages were calculated without using correction factors or an internal standard. The Carbon-13 NMR technique allows the unequivocal identification of the main constituents of an essential oil to a content of 0.5-1.0% without any previous separation. The identification is based on comparison of the signals in the mixture spectrum with those of the reference spectra compiled in a laboratory spectral library, with the help of a laboratory-made software (17,18).

Analytical GC: The GC analyses were carried out using a Perkin Elmer autosystem GC apparatus equipped with two flame ionization detectors and fused capillary columns (50 m x 0.22 mm; film thickness 0.25 µm), BP-20 (polyethylene glycol) and BP-1 (dimethyl siloxane). The columns were temperature programmed from 60°C to 230°C (35 min) at 2°C/min. The carrier gas was helium (0.8 mL/min) and we used split mode: 1/60. Injector and detector temperatures were 280°C.

GC/MS: GC/MS analyses were performed by a Perkin Elmer quadripole MS system (model 910) coupled with the above gas chromatograph equipped with a BP-20 capillary column and operating under the same conditions as described above, except for the carrier gas flow rate (1 mL/min). The MS operating parameters were: ionization voltage 70 eV, ion source temperature 230°C, scan mass range 35-450 amu.

Carbon-13 NMR: All NMR spectra were recorded on a Bruker AC 200 Fourier Transform spectrometer operating at 50.323 MHz for ^{13}C, in deuterated chloroform, with all shifts referred to internal tetramethylsilane. Carbon-13 spectra were recorded with the following parameters: pulse width: 3.0 µs (flip angle 45°); acquisition time: 2.3 s for 32K data table with a spectral width of 12500 Hz (250 ppm); Composite Phase Decoupling mode; digital resolution: 0.763 Hz/pt. The number of accumulated scans was 5000-10000 for each sample (70 mg of the oil in 0.5 mL $CDCl_3$).

Results and Discussion

Most of the myrtle oils analyzed in the literature were commercial *i.e.* collective oils and exhibited several chemotypes. Even more, from few studies carried out on individual oils, compositional differences were observed on oils produced from plants harvested in different stations or at different stages of development (20). As a consequence, our work was divided in three parts:

- analysis of 8 collective samples supplied by local producers as a attempt to characterize the commercial myrtle oil of Corsica,
- analysis of 24 individual samples produced by hydrodistillation of plants collected everywhere all around the corsican coastline, in order to point out a chemical variability,
- analysis of 12 samples obtained from the same plant harvested each month, in order to determine if the composition is dependent on the stage of plant development.

Collective Oils: Identification of individual components was carried out by Carbon-13 NMR spectroscopy. The 8 samples (A-H) have the same qualitative composition (Table I). Corsican myrtle oil is characterized by a high content of α-pinene (48-59%) associated with 1,8-cineole (20-28%) and limonene (5-6%), as well as by low amounts of linalool or terpinen-4-ol and the lack of myrtenyl acetate. It appears obviously from our results, which corroborate the previous analyses of Garry and Chalchat (7), that Corsican myrtle oil has a chemical composition similar to Sardinian (11) and Tunisian (7) oils, but differs from the oil of Esterel, reported in the literature (8), by the lack of myrtenyl acetate.

Individual Oils: Analysis of 24 samples was performed by GC and GC/MS. Only the compounds which were present in more than 12 samples of the 24, were taken into account. The compositions of 3 representative samples (M1-M3) as well as, for each compound, the minimum, maximum and average contents are reported on Table II. The chemical compositions of individual samples exhibited only small qualitative differences. Nevertheless, significant variations were observed in the concentration of the main constituants. So, the contents of α-pinene and 1,8-cineole, which are always the major monoterpenes, ranged respectively from 42% to 66% and from 15% to 38%, instead of respectively 48-59% and 20-28% for the collective oils. Concurrently, the sum and the ratio of the contents of these two compounds, which seem to be negatively correlated (Figure 1), ranged from 64.8% to 83.0% and from 1.2 to 4.3, respectively. Also, the oils were always devoid of myrtenyl acetate. From these values it appears that all the 24 samples analyzed belong to the α-pinene/1,8-cineole chemotype; however, quantitative differences between the oils were found. For instance, the sample M2 had a composition close to the one of Tunisian (7) and Sardinian (11) oils as well as the commercial Corsican oils described above. The sample M1 could be compared to Lebanon and Iranian oils (close amounts of α-pinene and 1,8-cineole) (7,10). Finally, the sample M3 shows respectively a high and a reduced contents of α-pinene

Table II. Chemical composition (percentage) of individual samples of *Myrtus communis* from different localities all around the Corsican coastline*

Components	M1	M2	M3	Min.	Max.	Aver.
α-pinene	44.7	50.1	66.1	42.3	66.1	51.2
isobutyl isobutyrate	0.2	1.7	0.2	0.0	2.8	0.8
β-pinene	0.5	0.7	0.5	0.4	1.0	0.5
δ-3-carene		1.0			1.2	0.5
isobutyl 2-methylbutyrate	0.4	1.4			1.4	0.5
limonene	3.7	6.3	5.6	2.8	10.7	5.9
1.8-cineole	38.3	23.8	15.5	15.1	38.3	23.6
γ-terpinene	0.3	0.9			1.5	0.6
(E)-β-ocimene	0.3	1.3	0.4		1.7	0.3
p-cymene	0.5	1.3			2.6	1.0
terpinolene	0.7	2.0	0.5	0.3	2.2	1.1
linalool	2.6	3.6	1.7		4.5	2.4
α-terpineol	4.4	3.7	4.6	2.4	5.6	3.8
geranyl acetate	0.8	1.3	1.7	0.8	2.9	1.9
methyl eugenol	0.5		0.9		2.1	1.0
Total	97.9	99.1	97.7			

* the reported percentages are obtained by GC on the polar column

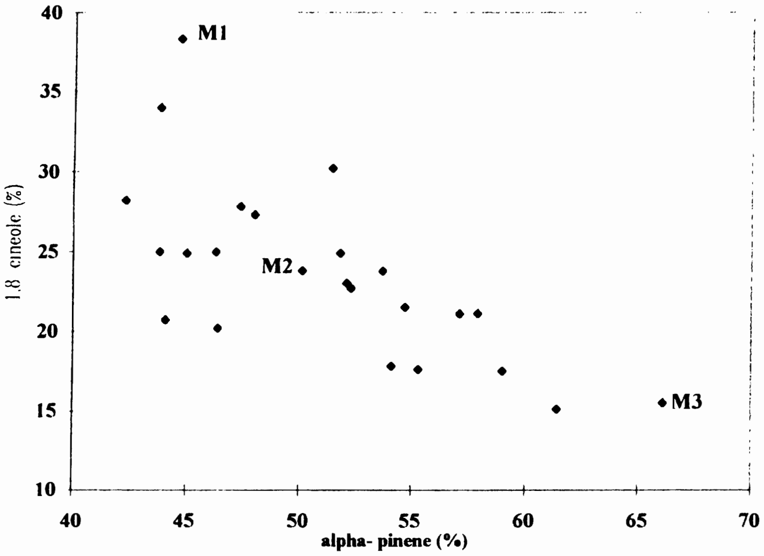

Figure 1. Percentages of 1,8-cineole vs α-pinene in individual oils of *Myrtus communis* (M1-M3, see text)

and 1,8-cineole, not yet reported (α-pinene/1,8-cineole = 4.3).

Composition Oil Evolution According to the Stage of Plant Development: The analysis of samples obtained by hydrodistillation of myrtle leaves and stems (and sometimes flowers and berries) of the same plant harvested each month during all the year, allowed us to determine the influence of the stage of plant development on the chemical composition of the oil. The contents of 9 main compounds identified by Carbon-13 NMR are reported in Table III. As can be seen α-pinene was always the major compound followed by 1,8-cineole and limonene. Otherwise, the vegetative state had a large

Table III. Chemical composition (percentage) evolution of an individual oil of *Myrtus communis*

	Period of distillation													
	1993									1994				
	1	2	3	4	5	6	7	8	9	10	11	12		
Components	**Feb**	**Mar**	**Apr**	**May**	**Jun**	**Jul**	**Sep**	**Oct**	**Nov**	**Dec**	**Jan**	**Feb**	**Min**	**Max**
α-pinene	48.8	51.2	47.4	57.2	53.0	51.3	56.9	47.0	51.8	51.7	50.0	56.2	47.0	57.2
δ-3-carene	1.1	0.7	0.8	0.8	1.2	0.9	1.0	0.9	0.9	0.6	0.7	1.0	0.6	1.2
limonene	6.3	5.8	6.0	5.9	5.7	5.7	5.8	6.0	5.7	4.8	5.5	5.5	4.8	6.3
1,8-cineole	23.2	24.7	29.6	20.5	17.3	21.1	17.0	20.6	17.1	12.0	23.9	16.9	12.0	29.6
p-cymene	2.6	2.0	2.0	2.8	0.9	1.5	2.5	3.3	3.1	3.4	1.7	2.5	0.9	3.4
terpinolene	1.1	0.8	0.8		2.7	1.4	0.7	0.3	0.4	0.7	1.0	1.0	0.3	2.7
linalool	2.0	1.8	1.9	1.6	2.3	2.5	2.1	2.4	2.0	2.5	2.0	3.1	1.6	3.1
α-terpineol	2.7	2.4	3.0	2.6	4.6	4.0	2.3	2.0	1.8	1.6	1.8	1.8	1.6	4.6
geranyl acetate	1.4	1.4	1.2	0.8	0.8	1.6	2.1	1.8	1.2	1.4	1.9	1.7	0.8	2.1
Total	89.2	90.8	92.7	91.4	88.5	90.0	90.4	86.4	85.0	79.6	88.5	89.8	79.6	91.9
Yield (%)	0.25	0.30	0.32	0.24	0.38	0.37	0.35	0.44	0.35	0.29	0.28	0.25	0.24	0.44

* the reported percentages are obtained by GC on the polar column

influence on the yield of hydrodistillation which ranged from 0.24% to 0.44%. Therefore, the yield seems to be the first parameter to take account if commercial production of the oil is to be performed. Also, it should be noted that the June-November period combined good yields and a relatively constant chemical composition, consequently, it is the recommended harvest time for commercial oil production in Corsica.

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