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Chemical variability of *Juniperus oxycedrus* ssp. *oxycedrus* berry and leaf oils from Corsica, analysed by combination of GC, GC–MS and ^{13}C-NMR

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ABSTRACT: The composition of 27 samples of berry oil and 54 samples of leaf oil of *Juniperus oxycedrus* ssp. *oxycedrus* from Corsica was investigated by GC, GC–MS and ^{13}C-NMR. The main constituents were terpene hydrocarbons, especially α-pinene, myrcene and germacrene D in the oils from berries and α-pinene, β-phellandrene and Δ3-carene in the oils from leaves. The results of the analyses were submitted to *k*-means partitioning and principal component analysis, which allowed the distinction of two compositions in the berry oils (differentiated by the contents of α-pinene, myrcene and germacrene D) and in the leaf oils (differentiated by the contents of α-pinene, β-phellandrene and Δ3-carene). (*Z*)-6-Pentadecen-2-one, an unusual alkenone present in several samples of leaf oil, was identified using ^{13}C-NMR spectroscopy.

KEY WORDS: *Juniperus oxycedrus* ssp. *oxycedrus*; Cupressaceae; essential oil; GC–MS; ^{13}C-NMR; Infraspecific variability; (*Z*)-6-pentadecen-2-one

Introduction

The genus *Juniperus* belongs to the family Cupressaceae, division Gymnospermeae. *Juniperus oxycedrus* L. comprises three subspecies, *J. oxycedrus* ssp. *oxycedrus*, *J. oxycedrus* ssp. *macrocarpa* and *J. oxycedrus* ssp. *badia*, endemic to Spain.[1] *J. oxycedrus* ssp. *oxycedrus*, which is the subject of the present study, is a common evergreen shrub or small tree. It grows wild throughout all the Mediterranean regions to northern Iran on rocky and sunny places, on dry hills and mountainous tracts, up to 1900 m altitude.[2] In Corsica, it is found from coastal zones to the mountains of the centre of the island (1000 m altitude).[1,3]

J. oxycedrus was utilized since the antiquity to produce a sesquiterpene-rich oil, named 'empyreumatic' oil, or 'cade oil', used in dermatology and cosmetology.[4,5] Otherwise, several studies reported on the chemical composition of solvent extracts from wood,[5] berries[6] and leaves[7] of *J. oxycedrus*, in which sesquiterpenes, diterpene acids and polyphenolic compounds have been isolated and characterized. Similarly, the essential oil of *J. oxycedrus* is obtained by hydrodistillation of leaves, berries or wood. Although these oils are usually characterized by a high content of α-pinene, whatever the subspecies and the origin, several compositions could be distinguished according to the content of the other abundant components. For instance, concerning leaf oils, the reported compositions are: (a) for *J. oxycedrus* ssp. *oxycedrus* (or for subspecies not specified), α-pinene (Portugal,[8] Sardinia,[9] and Croatia[10]) α-pinene/Δ-3-carene (Portugal,[8] Spain[11]); α-pinene/limonene (Italy,[12] Greece[11]); α-pinene/limonene/α-terpinyl acetate/β-caryophyllene (Italy[13]); α-pinene/β-phellandrene/terpinolene (Greece[14]), germacrene D and manoyl oxide (supercritical CO_2 extract, Sardinia[15]); (b) for the subspecies *macrocarpa*, α-pinene (Italy[12,13]), α-pinene/α-terpineol (Italy[12]); α-pinene/sabinene (Spain[11]); (c) for *J. oxycedrus* ssp. *badia*, α-pinene/germacrene D/manoyl oxide (Spain[11]).

The compositions of the berry oils were dominated by: (a) α-pinene alone (ssp. *macrocarpa* from Italy,[12,13] ssp. *oxycedrus* from Portugal[8] and from Italy;[15] subspecies not specified from Croatia,[10] Greece[16] and from Spain[17]); (b) α-pinene/myrcene (Italy[12,13]); (c) myrcene/α-pinene/γ-cadinene (Crete[18]), or myrcene/citronellol/α-pinene (Greece[19]); (d) sesquiterpenes (*J. oxycedrus*, Spain[20]); (e) germacrene D/α-pinene/myrcene (supercritical CO_2 extract, Sardinia[15]).

J. oxycedrus wood oil has been studied less frequently. It is a sesquiterpene-rich oil dominated either by δ-cadinene/epi-cubenol (Spain[21]) or by δ- and γ-cadinenes/calamenene/cubenol/β-maaliene (France).[5]

It obviously appears from the literature that *J. oxycedrus* leaf and berry oils exhibited a chemical variability. The purpose of this study was to characterize the

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Corsican *J. oxycedrus* ssp. *oxycedrus* through the chemical composition of leaf and berry oils and to investigate if chemical variability occurred in insular population. During the completion of that work, we were confronted with the identification of an unusual alkenone which was carried out by NMR.

Experimental

Plant Material and Isolation Procedure

Fifty-four samples of leaves and 27 samples of berries of the same shrubs were collected during the period May–July 2003, in the area of distribution of *J. oxycedrus* ssp. *oxycedrus* at different altitudes (Figure 1): Rocapina, littoral (nine samples of leaves, L1–9, one sample of berries, B2); Porto, littoral (10 samples of leaves, L21–30, six samples of berries, B22, B23, B25, B26, B29, B30); Osani, 200 m (11 samples of leaves, L10–20, six samples of berries, B11, B12, B17–20); Corte, 400 m (12 samples of leaves, L31–42; eight samples of berries, B31–38); and Restonica Valley, 900 m (12 samples of leaves, L43–54, six samples of berries, B43, B44, B46, B48–50).

Berries and leaves were submitted to hydrodistillation for 3 h using a Clevenger-type apparatus. Essential oil yields were in the range 0.04–0.26% (w/w) for fresh leaves, 0.28–1.53% (w/w) for fresh berries.

Analytical GC

GC analysis was carried out with a Perkin-Elmer Autosystem apparatus equipped with two flame ionization detectors, and fused-silica capillary columns (50 m × 0.22 mm i.d., film thickness 0.25 μm), BP-1 (polydimethylsiloxane) and BP-20 (polyethyleneglycol). The oven temperature was programmed from 60 °C to 220 °C at 2 °C/min and then held isothermal for 20 min; detector temperature, 250 °C; injector temperature, 250 °C; injection mode, split 1:60; carrier gas, helium at 0.8 ml/min; injected volume, 0.5 μl solution of 50 μl of the mixture (oil or fraction of chromatograph) diluted in 350 μl CCl_4.

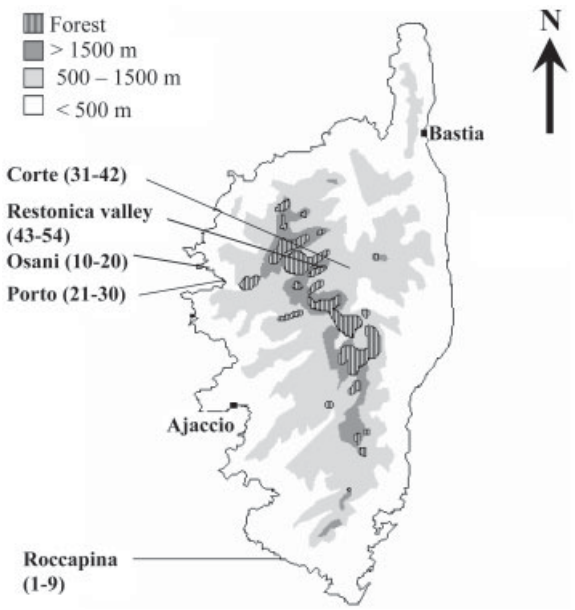

Figure 1. Sampling of *Juniperus oxycedrus* ssp. *oxycedrus* from Corsica

GC–MS Analysis

GC–MS analysis was performed with a Hewlett-Packard 6890 gas chromatograph, equipped with a HP1 fused-silica column (polydimethylsiloxane, 30 m × 0.25 mm i.d., film thickness 0.25 μm) and interfaced with a Hewlett-Packard Mass Selective Detector 5973 (HP Enhanced ChemStation software, version A.03.00). Oven temperature program, 70 °C to 220 °C at 3 °C/min, then held at 220 °C for 15 min; injector temperature, 250 °C; carrier gas, helium, adjusted to a linear velocity of 30 cm/s; split ratio, 1:40; interface temperature, 250 °C; MS source temperature, 230 °C; MS quadrupole temperature, 150 °C; ionization energy, 70 eV; ionization current, 60 μA; scan range, 35–350 u.

^{13}C-NMR Analysis

All NMR spectra were recorded on a Bruker AC 200 Fourier transform spectrometer operating at 50.323 MHz for ^{13}C, equipped with a 10 mm (or 5 mm) probe, in $CDCl_3$, with all shifts referred to internal TMS. ^{13}C-NMR spectra were recorded with the following parameters: pulse width (PW), 5 μs (or 3 μs) (flip angle, 45°); acquisition time, 1.3 s for 32 K data table with a spectral width (SW) of 12 500 Hz (250 ppm); CPD mode decoupling; digital resolution 0.763 Hz/pt. In a typical procedure, 200 mg (or 70 mg) of the mixture (essential oil and fractions of chromatography) were diluted in 2 ml (or 0.5 ml) $CDCl_3$. The number of accumulated scans ranged between 2000 and 10 000 for each sample, depending on the available amount of product. Exponential line broadening multiplication (LB = 1 Hz) of the free induction decay (FID) was applied before Fourier transformation.

Identification of Components

Identification of the individual components was based on: (a) comparison of their GC retention indices (RI) on apolar and polar columns, determined relative to the retention times of a series of *n*-alkanes with linear interpolation (Target Compounds software of Perkin-Elmer), with those of authentic compounds or literature data; (b) computer matching with a laboratory made mass spectral library and commercial libraries,[22,23] and comparison of spectra with literature data;[24,25] (c) comparison of the signals in the ^{13}C-NMR spectra of the selected samples with those of reference spectra compiled in the laboratory spectral library, with the help of laboratory-made software.[26,27] All the samples were submitted to chromatographic analysis with two columns of different polarity. On the basis of their chromatographic profile, seven samples of leaf oil and three samples of berry oil were analysed by GC–MS, and 27 samples of leaf oil

and 15 samples of berry oil were analysed by ^{13}C-NMR. Each component was identified by MS and/or ^{13}C-NMR in at least four samples of leaf oil and four samples of berry oil.

Identification of (*Z*)-6-pentadecen-2-one (52)

In some samples, the component **52** (RI = 1647 and 2030 on apolar and polar columns, respectively) was 'tentatively' identified by MS as dodecadienyl acetate (M = 224). This suggestion was ruled out by analysis of the ^{13}C-NMR spectrum of a sample of leaf oil (L18) where compound **52** accounted for 5.4%. The chemical shift values, remaining after removal of the values of the identified components, suggested the occurrence of a double bond and a methyl ketone. Consequently sample L18 (600 mg) was chromatographed on a silica gel column (200–500 µm) and three fractions (F1–F3) were eluted respectively with pentane, pentane:diethyl oxide 95:05 and diethyl oxide. Compound **52** accounted for 50% in fraction F2 (beside minor components) and it was possible to observe all its signals in the ^{13}C-NMR spectrum of the fraction. From the chemical shift values and DEPT spectrum, it appeared that compound **52** was a 2-pentadecenone (M = 224, in agreement with the mass spectrum) exhibiting the (*Z*) stereochemistry of the double bond (signals of allylic methylenes at 26.51 and 27.26 ppm). The position of the double bond was ensured by the examination of the lanthanide-induced shift (LIS) on the signals of all the carbons. Four equimolar increments of Yb(fod)$_3$ were added to a solution of the substrate in $CDCl_3$, the molar ratio [Yb(fod)$_3$]:[substrate] ranging from 0 to 0.3. For each carbon we quantified the LIS values ($\Delta\delta$, ppm) as a function of the [lanthanide complex]:[substrate] ratio by measuring the slopes of the lines $\Delta\delta = [f(\text{Yb(fod)}_3)]/[\text{substrate}]$ (Figure 2). Consequently, the compound is (*Z*)-6-pentadecen-2-one.[28]

^{13}C-NMR of (*Z*)-6-pentadecen-2-one: δ 29.89 (C-1), 209.39 (C-2), 43.06 (C-3), 23.73 (C-4), 26.51 (C-5), 128.59 (C-6), 131.10 (C-7), 27.26 (C-8), 29.75 (C-9), 29.35 (C-10), 29.55 (C-11), 29.35 (C-12), 31.93 (C-13), 22.70 (C-14), 14.13 (C-15).

MS *m/z* (rel. int.): 224 [M$^+$] (8), 166 (33), 138 (33), 125 (22), 124 (21), 123 (10), 111 (18), 110 (23), 109 (23), 97 (24), 96 (72), 95 (38), 84 (10), 83 (23), 82 (77), 81 (61), 80 (13), 79 (18), 71 (26), 69 (37), 68 (65), 67 (65), 58 (16), 57 (18), 55 (42), 54 (67), 43 (100), 41 (42), 39 (11).

Data Analysis

Principal components analysis (PCA) was performed using Statgraphics Plus 2.1 (Uniwin plus, France); *k*-means clustering was performed using a *k*-means partitioning program (Pierre Legendre, Canada).[29]

Results and Discussion

We collected leaves and berries on individual plants from locations covering the geographic range of *J. oxycedrus* ssp. *oxycedrus* in Corsica (Figure 1).

Fifty four-components, amounting to 98.7–86.0% of the leaf oil and 33 components amounting to 99.0–91.9% of the berry oil were identified (Table 1). We found in total 35 monoterpenes, 18 sesquiterpenes, three diterpenes (manoyl oxide, abietatriene and abietadiene) and one acyclic 'ketoalkene', (*Z*)-6-pentadecen-2-one. All the identified components were taken into account for statistical analysis (PCA and *k*-means analysis).

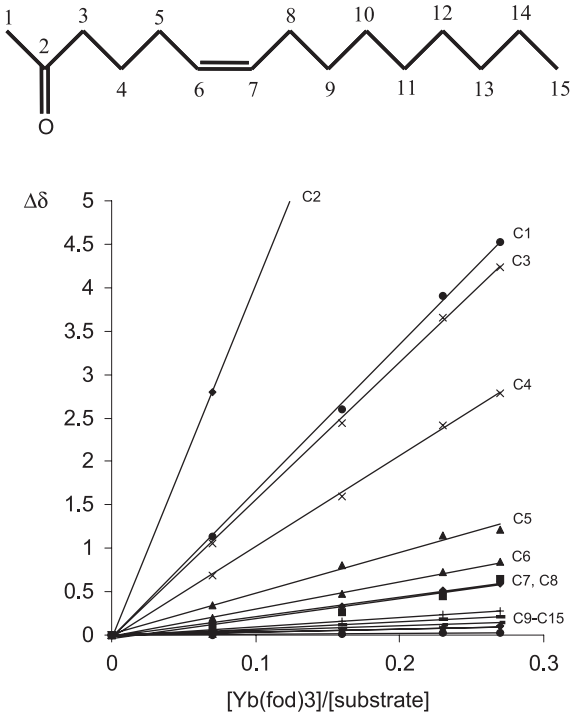

Figure 2. Identification of (*Z*)-6-pentadecen-2-one (see experimental)

Berry Oil

PCA combined with *k*-means suggested the occurrence of a chemical variability. Within the population of *J. oxycedrus* ssp. *oxycedrus* berry oil, the partition of the samples into two groups was the best one proposed by the *k*-means partitioning program. In the PCA the first two axes accounted for 80% and 18%, respectively (Figure 3). The two groups were distinguished on the basis of α-pinene, myrcene and germacrene D contents.

In the oils of group I (56% of the samples), characterized by α-pinene (58.7%, SD = 5.9), myrcene (14.1%, SD = 5.5) and germacrene D (11.7%, SD = 4.4), the ratio of the three components is close to 5:1:1, whereas in the oils of group II (44% of the samples), also characterized by α-pinene (41.1%, SD = 6.0), myrcene (21.9%, SD = 4.5) and germacrene D (19.9%, SD = 5.7), the ratio of the same three compounds is close to 2:1:1. The remaining components are present in very low amounts (e.g. maximum for limonene in group II was 1.7%).

We observed that in each location, the distribution of the two groups was heterogeneous. For instance, two

Table 1. Chemical composition of berry and leaf oils of *Juniperus oxycedrus* ssp. *oxycedrus* from Corsica

No.		BP-1	BP-20	Berry oil				Leaf oil			
				Group I		Group II		Group I		Group II	
				Mean	SD	Mean	SD	Mean	SD	Mean	SD
1	Tricyclene	919	1007	0.1	0.1	—	—	0.2	#	0.2	#
2	α-Pinene	930	1022	58.7	5.9	41.1	6.0	73.3	5.8	56.1	5.7
3	α-Fenchene	940	1052	0.1	0.1	0.1	0.1	0.2	0.2	0.5	0.3
4	Camphene	943	1060	0.2	0.1	0.2	0.1	0.3	0.1	0.3	0.1
5	Verbenene	945	1120	—	—	—	—	0.1	0.1	0.2	0.2
6	Sabinene	962	1116	0.3	0.1	0.3	#	0.2	0.1	0.2	0.1
7	β-Pinene	967	1105	1.5	0.5	1.1	0.5	1.6	0.4	1.5	0.3
8	Myrcene	977	1155	14.1	5.5	21.9	4.5	2.3	0.7	2.3	0.6
9	α-Phellandrene	994	1160	—	—	—	—	0.7	0.8	0.9	0.8
10	Δ3-Carene	1002	1144	—	—	—	—	0.7	1.9	8.2	7.5
11	*p*-Cymene	1008	1264	—	—	—	—	0.7	0.6	1.3	0.8
12	Limonene*	1020	1195	1.4	0.4	1.7	0.2	1.2	0.3	1.3	0.5
13	β-Phellandrene*	1020	1207	0.3	0.1	0.3	0.1	3.3	3.6	4.2	3.0
14	γ-Terpinene	1047	1244	0.1	#	—	—	0.1	#	0.1	#
15	*p*-Cymenene	1072	1432	—	—	—	—	0.1	0.1	0.2	0.1
16	Terpinolene	1074	1275	0.6	0.1	0.5	0.2	0.4	0.2	0.7	0.4
17	Linalol	1080	1539	—	—	—	—	0.1	0.1	0.1	0.1
18	α-Campholenal	1100	1482	—	—	—	—	0.6	0.4	0.8	0.6
19	Camphor	1115	1517	0.2	0.2	0.1	0.1	0.1	0.1	0.1	0.1
20	*trans*-Pinocarveol	1119	1644	—	—	—	—	0.6	0.4	0.9	0.6
21	*cis*-Verbenol	1123	1657	—	—	—	—	0.2	0.2	0.3	0.2
22	*trans*-Verbenol	1125	1666	—	—	—	—	0.2	0.2	0.4	0.4
23	*trans*-Pinocamphone	1135	1507	—	—	—	—	0.2	0.2	0.3	0.1
24	*p*-Mentha-1,5-dien-8-ol	1140	1714	—	—	—	—	0.5	0.4	0.9	0.6
25	Borneol	1150	1696	0.1	0.2	0.1	0.1	0.1	0.1	0.2	0.1
26	Cryptone	1153	1660	—	—	—	—	0.1	0.1	0.2	0.1
27	*p*-Cymene-8-ol*	1156	1802	—	—	—	—	0.1	0.1	0.2	0.1
28	Terpinen-4-ol*	1156	1592	0.3	0.1	0.2	0.1	0.2	#	0.2	0.2
29	Myrtenal	1165	1619	—	—	—	—	0.2	0.1	0.3	0.2
30	α-Terpineol	1168	1684	0.4	0.3	0.2	0.2	0.5	0.2	0.7	0.4
31	Myrtenol	1176	1782	—	—	—	—	0.2	0.1	0.3	0.2
32	Verbenone	1178	1710	—	—	—	—	0.2	0.1	0.4	0.6
33	*trans*-Carveol	1192	1828	—	—	—	—	0.2	0.2	0.4	0.3
34	Bornyl acetate	1262	1570	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2
35	α-Terpinyl acetate	1324	1684	—	—	—	—	0.2	0.4	0.3	0.5
36	α-Cubebene	1350	1453	0.6	0.3	0.3	0.2	—	—	—	—
37	α-Copaene	1378	1488	0.2	0.1	0.1	0.1	0.1	0.1	0.1	#
38	β-Elemene	1388	1588	—	—	—	—	0.1	0.1	0.1	0.1
39	(*E*)-β-Caryophyllene	1412	1585	0.8	0.3	1.3	0.2	0.2	0.1	0.2	0.1
40	α-Humulene	1445	1675	0.8	0.3	1.2	0.2	0.2	0.1	0.2	0.1
41	(*E*)-β-Farnesene	1448	1662	0.3	0.1	0.4	0.1	—	—	—	—
42	γ-Muurolene	1456	1666	0.5	0.2	0.6	0.2	0.2	0.1	0.1	0.1
43	Germacrene D	1477	1701	11.7	4.4	19.9	5.7	1.5	0.8	1.2	0.8
44	α-Muurolene	1496	1717	0.3	0.1	0.3	0.1	—	—	—	—
45	γ-Cadinene	1502	1748	0.9	0.6	1.2	0.6	0.3	0.2	0.3	0.3
46	δ-Cadinene	1508	1745	0.8	0.3	1.0	0.3	0.3	0.1	0.2	0.2
47	(*E*)-Nerolidol	1541	2033	0.2	0.4	0.2	0.6	0.4	0.7	0.4	0.8
48	Caryophyllene oxide	1566	1989	—	—	—	—	0.3	0.2	0.4	0.2
49	Humulene oxide	1600	2040	—	—	—	—	0.1	#	0.1	0.1
50	τ-Cadinol	1620	2161	0.3	0.2	0.4	0.2	0.2	0.1	0.2	0.2
51	α-Cadinol	1631	2219	0.2	0.2	0.2	0.2	0.3	0.1	0.4	0.3
52	(*Z*)-6-Pentadecen-2-one	1647	2030	—	—	—	—	0.7	1.0	1.1	0.9
53	(*E*,*E*)-Farnesol	1690	2343	—	—	—	—	0.4	0.4	0.6	0.7
54	(*E*,*E*)-Farnesal	1706	2255	—	—	—	—	0.1	0.1	0.2	0.1
55	Manoyl oxide	1985	2347	0.4	0.3	0.3	0.2	0.3	0.4	0.5	0.6
56	Abietatriene	2034	2488	0.1	0.1	0.1	0.1	0.2	0.1	0.4	0.3
57	Abietadiene	2071	2450	0.8	0.6	0.3	0.2	0.4	0.4	1.2	1.2

Order of elution and percentages are given on apolar column (BP-1), except for compounds with an asterisk (*), percentages on polar column (BP-20). #SD inferior to 0.05.

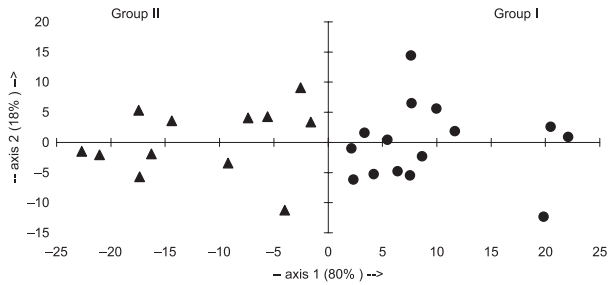

Figure 3. PCA scatterplot of 27 samples of *Juniperus oxycedrus* ssp. *oxycedrus* berry oil from Corsica

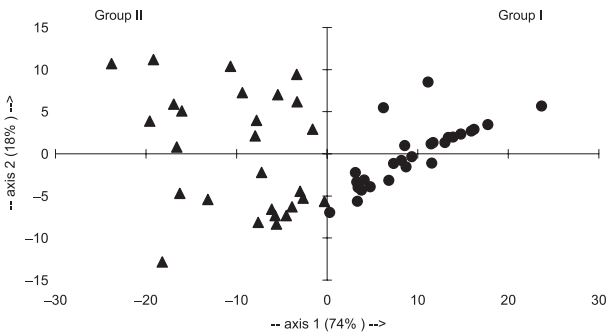

Figure 4. PCA scatterplot of 54 samples of *Juniperus oxycedrus* ssp. *oxycedrus* leaf oil from Corsica

locations were distinguished by a clear dominance of one group, group I for Corte (all the samples) and group II for Restonica Valley (5/6 samples). Conversely, the samples from Osani exhibited a ratio of group I:group II = 1:2, while the samples from Porto were equally divided in the two groups.

Although most of the berry oils of *J. oxycedrus* ssp. *oxycedrus* reported in the literature are dominated by α-pinene and myrcene, the ratio of the two compounds may vary drastically. Moreover, oils of different origins could be differentiated by the occurrence of other components in appreciable contents. Indeed, the berry oil of *J. oxycedrus* ssp. *oxycedrus* from Corsica, which also contained α-pinene and myrcene as the major components, could be distinguished from oils of other countries by the presence of germacrene D in high contents. For instance, γ-cadinene (15.7–21.2%) was the third most abundant compound in the oils from Greece.[18] Two other Greek oils of *J. oxycedrus* were characterized by the occurrence of citronellol (16.3%, 26.8%) besides myrcene (23.4%, 24.3%) and α-pinene (16.7%, 14.4%).[19] The α-pinene-rich oil (66.3% and 61.2%) from Croatia[10], as well as the α-pinene/myrcene-rich oils (27.0/28.4% and 31.6/40.4%,) from Italy,[12,13] contained germacrene D in very low contents. Finally, the Corsican oil obviously differed from the sesquiterpene-rich oil of *J. oxycedrus* from Spain.[11] Similarly, it could be pointed out that two berry oils from the subspecies *macrocarpa*, reported as α-pinene-rich oils (85.1%, Italy;[12] 63.0%, Greece[16]), contained only very small quantities of germacrene D (<0.3%). Conversely, the oil from Corsica is similar to that from Portugal[8] (α-pinene, 46.7–74.3%; myrcene, 1.5–18.3%; and germacrene D, 1.2–19.4%). It could be pointed out that germacrene D (13.8%) is the major component of the supercritical CO_2 extract from Sardinia.[15]

Leaf Oil

Concerning the leaf oil, once again, PCA combined with *k*-means analysis suggested the existence of two principal clusters within this oil. The first two axes in the PCA accounted for 74% and 18%, respectively (Figure 4). In the samples of group I (50% of the samples), α-pinene largely dominated the chemical composition (mean = 73.3%, SD = 5.8), whereas the amount of the other components did not exceed 3.3% each (β-phellandrene). α-Pinene is again the major component in the samples of group II, but in lower amounts (mean = 56.1%, SD = 5.7) accompanied by Δ-3-carene (8.2%, SD = 7.5, whereas this product was present at lower content in the group I) and β-phellandrene (4.2%, SD = 3.0).

So, the leaf oil of *J. oxycedrus* ssp. *oxycedrus* from Corsica exhibited a chemical variability, with two composition patterns, α-pinene and α-pinene/Δ-3-carene. Both composition patterns are present in all the stations, located at different altitudes, with ratios of group I:group II varying from 1.5:1 to 1:2.

The mean chemical compositions of the groups I and II differed from those of other countries: (a) Italy (ssp. *oxycedrus*), α-pinene and limonene (26.3% and 30.0%);[12] (b) Italy (ssp. *oxycedrus*), limonene/α-terpinyl acetate/α-pinene/β-caryophyllene (12.3/9.5/8.1/7.1%)[13]; (c) Greece, α-pinene (2.3–56.6%), accompanied by β-phellandrene (6.8–52.6%) and terpinolene (0.1–22.7%)[14] (subspecies not specified); (d) Croatia (*J. oxycedrus*), α-pinene (41.4%) followed by manoyl oxide (12.3%);[10] (e) Italy, supercritical CO_2 extract, germacrene D (15.9%) and manoyl oxide (10.2%).[15] The oils of Corsica differed also from most of the oils reported recently by Adams, who distinguished the leaf oils of three subspecies of *J. oxycedrus* (ssp. *oxycedrus*, *badia* and *macrocarpa*), all dominated by α-pinene (25–43%), by the presence of limonene (4.5–28%) for the subspecies *oxycedrus* (Spain and Greece), germacrene D (3.4–24.5%) and variable amounts of manoyl oxide (0.2–21%) for the subspecies *badia* (Spain) and sabinene (26.5%) for the subspecies *macrocarpa* (Spain).[11]

Conversely, the compositions of groups I and II from Corsica corresponded to the two reported compositions of *J. oxycedrus* ssp. *oxycedrus* oils from Portugal: α-pinene (mean = 78.8%) and α-pinene/Δ-3-carene (mean = 65.4/10.7%).[8] Otherwise, the composition of the group I, largely dominated by α-pinene, is similar to that of the

oil of the subspecies *macrocarpa* from Italy (α-pinene = 81.3% and 75.5%).[12]

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