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Chemical composition of essential oils of *Juniperus communis* L. obtained by hydrodistillation and microwave-assisted hydrodistillation

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Abstract
Jiniperus communuis L. essential oils obtained by hydrodistillation (HD) and microwave-assisted hydrodistillation (MAHD) were investigated by capillary gas chromatography and gas chromatography/ mass spectrometry. Both distillation methods and analytical results were compared. Among the identified volatile compounds from *J. communis*, the main components from both oils were found to be similar, but their contents were significantly different including α-pinene (14.2, 9.7%) followed by sabinene (12.4, 16.1%), γ-terpinene (5.9, 1.7%), terpinene 4-ol (14.1, 9.1%), (*Z*,*Z*)-farnesol (5.4, 6.6%) and manoyl oxide (4.1, 11.7%), respectively. Microwave-assisted hydrodistillation appears to be an effective method for the production of essential oils.

Keywords: Juniperus communis; leaves; Cupressaceae; essential oil composition; microwave-assisted hydrodistillation.

1. Introduction
The genus *Juniperus* consists of approximately 75 species divided into 3 sections; *Caryocedrus* (one species, *J. drupacea* Labill); *Juniperus* (syn: sect. *Oxycedrus* content 14 species) and *Sabina* (approximately 60 species). *J. communis* is the only *Juniperus* species that occurs in both hemispheres [1]. The flora of Algeria lists five native *Juniperus* species namely, *J. communis, J. phoenicea, J. oxycedrus, J. sabina* and *J. thurifera* [2]. *Juniperus communis* L., (common juniper) is an evergreen shrub or small tree (1-3m high). It grows wild in many parts of Europe, Asia, North America and North Africa with temperate or cold climate [3]. In folk and official medicine, *J. communis* possesses wide range pharmacological activities such as antifertility [4], hypoglycemic [5], anidiabetic [6], diuretic, antiseptic, carminative, stomachic and antirheumatic [7-10]. In addition, the essential oil, infusions, decoctions and alcoholic extracts of *J. communis* berries are used in different fields (pharmaceuticals, food industry, perfumery and cosmetics) [8-12].

The chemical composition of essential oils obtained by hydrodistillation from juniper leaf (needles) [13-28] and berries [3,8,10,19,22,29-36] have been studied by several authors. α-Pinene dominated in the most leaf oils of *J. communis* growing in Sweden [7], Greece [2], Croatia [18], France [16], Lithuania [21, 22], Estonia [24], China [25] and Bulgaria [26]. However, sabinene dominated only in several leaf oils from Italy [13, 15], India [27], north Iran [19] and Morocco [28].

Several extraction methods are available for obtaining essential oil components from plant materials. The composition of the oils isolated from the berries or needles of juniper by solvent distillation (SDE) and supercritical fluid extractions (SFE), also the analysis technique (HS-SPME) are studied [3,10,20,34,37-40]. Microwave extraction (MW) has gained increasing attention in the recovery of essential oils (EOs). Microwave–assisted hydrodistillation (MWHD) method is a more recent technique used to recover volatile components [41-43]. Heat is produced by microwave energy. The sample reaches its boiling point very rapidly, leading to a very short extraction. In addition, with the microwave distillation technique it is possible to achieve distillation with the indigenous water of the fresh plant material.

Recently, In Bouira city of Northern Algeria (National Park of Djurdjura), Foudil-Cherif and Yassaa studied enantiomeric and non-enatiomeric distribution of monoterpenes in the headspace of *J. Communis,* by the use of

HS-SPME and chiral-GC/MS [44].To the best of our knowledge, there is no previous report on the microwave extraction of *J. communis* leaf essential oil. The aim of this paper was to investigate the chemical composition of essential oils from needles of *J. communis* from Algeria obtained by hydrosistillation (HD) and microwave-assisted hydrodistillation (MAHD).

2. Materials and methods

2.1 Plant Material. *Juniperus communis* was collected in May 2008 from Sidi-Lakhdar City, Mostaghanem Province-West Algerian. The plant was authenticated in the botanical department, National Institute of Agronomic (NIA, Algeria), where a voucher specimen of the plant has been placed in the Herbarium (HNIA/FA/N°: P101bis) of this school.

2.2 **Essential oil isolation**

2.2.1 Hydrodistillation **(HD).** The shade-dried and finely powdered leaves of the plant were hydrodistilled for three hours using a Clevenger-type apparatus. The essential oil was dried over anhydrous sodium sulphate, filtered and stored in the dark at 4°C until analysis.

2.2.2

2.2.2 Microwave–assisted hydrodistillation. Microwave–assisted hydrodistillation (MWHD) was performed at atmospheric pressure using a microwave laboratory oven operating at 2450MHz (Model: Midea AG823ABI). 100g of dried needles of *J. communis* were heated using a fixed power of 800W for 30min with the addition of 50 mL distilled water. The extraction oil was continues at 100°C until no more essential oil was obtained. The essential oil was collected, dried under anhydrous sodium sulphate, filtred and stored at +4°C until used.

2.3 Analytical procedure

2.3.1. Gas Chromatography (GC-FID)

GC-FID analyses were carried out using a Shimadzu GC-17A V.3 chromatograph using fused silica capillary columns with two different stationary phases: DB-5 and HP20M. The various parameters fixed for the DB-5 column are: 30 m x 0.32 mm, 0.25 µm film thickness. The temperature program was 60°C for 3 min then 3°C/min to 240°C for 3 min; injector 250°C; detector 250°C; N_2 was used as carrier gas at a flow rate 1 mL/min in the split mode 1:50, with an injection volume of 0.2 µL. For the HP20M the parameters were (50m x 0.32mm, 0.25µm film thickness. Column temperature program was 50°C for 3min, there 2°C/min to 220°C for 5min; other parameters were the same as with DB-5 column. Quantitative data was obtained from electronic integration of area percentages without the use of correction factors. In order to determine retentions indices (RI), a series of n-alkane (C_5-C_{28}) mixtures were analyzed under the same operative conditions on DB-5 and HP-20M column; the samples indices were calculated following Van den Dool and Kratz [45].

2.3.2. Gas Chromatography-Mass Spectrometry (GC-MS)

The GC-MS analysis was performed on a TRACE GC Ultra-DSQ II mass spectrometer using a DB-5 capillary column (30 m x 0.32 mm, 0.25 µm film thicknesses). It was programmed from 60°C (3min) to 240°C (3min) at 3°C/min with He carrier gas at a flow rate of 1 $mL.min^{-1}$ and injector heater 250°C. The MS conditions were EI source, electron energy 70 eV and source temperature 250°C. Acquisition mass range, m/z = 40-450.

2.4 Components Identification

Identification of the individual components was based on comparison of their GC retention indices (RI) on apolar and polar columns, with those of authentic compounds or literature data, and by comparing their mass spectral data with those stored in the spectrometer databases using the Nist, Wiley mass spectral libraries and comparison of spectra with literature data [46-49].

3. Results and discussion

Dried leaves of *J. communis* were hydrodistilled and microwave-assisted hydrodistilled to yield volatile compounds. The essential oils were analysed by GC and GC/MS. Relative percentages of the characterized components calculated by FID integrator are given in Table1.

In HD and MWHD oils of *J. communis*, seventy four compounds representing 93.35% and 84.06% of the total oil were characterized, respectively. The results have shown that there was a difference in oil yields by both techniques, and similar oil profiles were obtained. Both oils were characterized by the main presence of α-pinene (14.2% and 9.7%), sabinene (12.4% and 16.1%), γ-terpinene (5.9% and 1.7%), terpinen-4-ol (14.1% and 1.9%), (*Z*,*Z*)-farnesol (5.4% and 6.6%) and manoyl oxide (4.1% and 11.7%), respectively. The total yield of the volatile fractions obtained through HD and MWHD was 0.26% and 0.14%, respectively. Higher amounts of oxygenated monoterpenes such as terpinen-4-ol, *trans*-pinocarveol, α-terpeneol (23.2%) were present in the oil of HD in comparison with MWHD (6.32%). However, HD oil contained more monoterpene hydrocarbons such as α-

thujene, α-pinene, sabinene, myrcene, α-terpinene, *p*-cymene, limonene, γ-terpinene and terpinolene (52.3%) than MWHD extracted oil (38.3%). A critical observation of the oil compositions revealed that higher amounts of diterpenes such as manoyl oxide, abietatrine, abietadiene, abietal and 4-epi-abietol (21.1%) are present in the essential oil isolated by MWHD in comparison with the oil extracted by HD (8.6%). This is probably due to the diminution of thermal and hydrolytic effects compared with hydrodistillation which uses a large quantity of water and is time and energy consuming. Water is a polar solvent, which accelerates many reactions that proceed via carbocation intermediates [43].

For comparison purposes, Table 2 summarizes previous studies on the volatile leaf oils from several populations of *J. communis* from different countries. Three main compounds and their percentages were only considered. According to the literature [13-28], the chemical pattern in the leaf oils of *J. communis* was to produce α-pinene (16.9-66.5%) as a major component. Our result confirmed this observation.

The juniper dried leaf oil from Croatia, containing α-pinene (16.9%), sabinene (12.1%) and terpinen-4-ol (7.7%) as the major constituents [18], is nearly similar to our oil with the main presence of terpinen-4-ol which was the second main compound in our study (Table 2).

Sabinene, one of the most important components (27.5 - 61.1%) in dried or fresh (no specified) leaf hydrodistilled oils from Italy (61.1%) [13], India (48.4%) [27], Northern Iran (40.7%) [19] and Morocco (27.5%) [28], and was the second main compound in corresponding dried leaf essential oils from Greece [14], Croatia [18], Estonia [24] and Bulgaria [26] (see Table 2). The dried leaf oil of *J. communis*, plant collected in National Botanical Garden of Iran (Tehran), is dominated by δ-3-carene (39.4%), β-phellandrene (25.1%) and α-pinene (24.5%) [20]. On the basis of the results summarized in table 2, we can classify the samples of *J. communis* three chemotypes the basis of major products of its essential oils leaves; chemotype α-pinene (North-East Lithuania, Vilnius District- Lithuania, Estonia, China, Sweden, Croatia, Greece, France and Algeria), chemotype sabinene (North Iran, India, Italy and Morocco) and chemotype δ-3-carene (Iran).

Table1. Essential oils components of needles of *J. communis* obtained by hydrodistillation (HD) and microwave-assisted hydrodistillation (MAHD)

N°	RI[a] DB5	RI[b] HP-20M	Compound[c]	HD[d]	MW-HD[d]	Methods of identification
1.	798	-	n-Hexenal	0.1	-	GC, GCMS
2.	846	1207	2-(*E*)-Hexenal	0.2	-	GC, GCMS
3.	920	1004	Tricyclene	0.06	-	GC, GCMS
4.	924	1021	α-Thujene	2.3	1.6	GC, GCMS
5.	933	1019	**α-Pinene**	**14.2**	**9.7**	GC, GCMS, CoGC[f]
6.	640	1053	α-Fenchene	tr	-	GC, GCMS
7.	945	1062	Camphene	0.2	0.1	GC, GCMS
8.	950	1075	Thuja-2,4(10)-diene	0.2	0.08	GC, GCMS
9.	971	1120	**Sabinene**	**12.4**	**16.1**	GC, GCMS
10.	973	1107	β-Pinene	0.5	0.2	GC, GCMS, CoGC
11.	981	-	*dehydro*-1,8-Cineol	tr[e]	-	GC, GCMS
12.	986	1155	Myrcene	2.9	1.4	GC, GCMS
13.	996	1126	δ-2-Carene	0.3	0.2	GC, GCMS
14.	1000	1172	α-Phellandrene	0.5	0.08	GC, GCMS
15.	1006	1142	δ-3-Carene	0.3	0.2	GC, GCMS, CoGC
16.	1012	1191	α-Terpinene	3.3	0.8	GC, GCMS, CoGC
17.	1019	1263	*p*-Cymene	2.2	3.0	GC, GCMS
18.	1024	1201	**Limonene**	**4.3**	**2.4**	GC, GCMS, CoGC
19.	1042	1233	*trans*-*β*-Ocimene	0.1	-	GC, GCMS
20.	1054	1239	**γ-Terpinene**	**5.9**	1.7	GC, GCMS

21.	1062	1416	*cis*-Sabinene hydrate	0.3	0.3	GC, GCMS
22.	1083	1276	Terpinolene	2.6	0.7	GC, GCMS, CoGC
23.	1093	1451	*trans*-Sabinene hydrate	0.4	0.3	GC, GCMS
24.	1111	1430	*trans*-Thujone	0.2	0.1	GC, GCMS
25.	1116	1479	*cis-p*-Menth 2-en-1-ol	0.6	0.1	GC, GCMS
26.	1120	1504	α-Campholenal	0.7	0.5	GC, GCMS
27.	1133	1659	*trans*-Pinocarveol	1.3	0.4	GC, GCMS
28.	1140	-	*trans*- Menth 2-en-1-ol	0.7	0.3	GC, GCMS
29.	1143	1709	*trans*-Verbenol	0.1	-	GC, GCMS
30.	1151	-	Sabina ketone	0.3	0.2	GC, GCMS
31.	1156	1628	Pinocarvone	0.1	-	GC, GCMS
32.	1162	1668	Borneol	0.6	0.2	GC, GCMS, CoGC
33.	1175	1590	**Terpinene 4-ol**	**14.1**	**1.9**	GC, GCMS, CoGC
34.	1179	-	*p*-Cymen-8-ol	0.4	0.2	GC, GCMS
35.	1185	1693	α-Terpineol	1.5	0.3	GC, GCMS, CoGC
36.	1189	-	Myrtenol	0.6	0.3	GC, GCMS
37.	1203	1648	*trans*-Piperitol	0.8	0.2	GC, GCMS
38.	1212	1831	*trans*-Carveol	0.5	0.2	GC, GCMS
39.	1221	1763	Citronellol	0.07	0.07	GC, GCMS, CoGC
40.	1230	-	Thymol, methyl ether	0.06	0.07	GC, GCMS
41.	1235	1757	Cumin-aldehyde	0.1	0.08	GC, GCMS
42.	1240	1637	Carvone	tr	-	GC, GCMS
43.	1243	-	Carvacrol, mrthyl ether	0.1	-	GC, GCMS
44.	1251	1593	Piperitone	0.2	0.1	GC, GCMS
45.	1273	-	*p*-Menth-1-en-7-al	0.1	0.4	GC, GCMS
46.	1281	-	α-Terpinene 7-al	0.1	0.1	GC, GCMS
47.	1292	-	*p*-Cymen-7-ol	0.1	0.2	GC, GCMS
48.	1304	-	Carvacrol	tr	-	GC, GCMS
49.	1373	1580	β-Bourbonene	0.07	0.1	GC, GCMS
50.	1380	1611	β-Elemene	tr	-	GC, GCMS
51.	1407	1613	β-Caryophyllene	0.09	0.2	GC, GCMS
52.	1441	1661	α-Humullene	0.07	0.08	GC, GCMS
53.	1465	1682	γ-Muurolene	tr	0.6	GC, GCMS
54.	1470	1704	Germacrene D	0.8	0.5	GC, GCMS
55.	1473	-	*trans-β*-Ionone	tr	0.2	GC, GCMS
56.	1511	1746	δ-Cadinene	0.2	0.2	GC, GCMS
57.	1557	2020	*trans*-Nerolidol	0.2	1.1	GC, GCMS
58.	1570	2102	Spathulenol	0.3	0.4	GC, GCMS
59.	1580	1964	Caryophyllene oxide	0.1	0.1	GC, GCMS
60.	1631	2195	*epi*-α-Murrolol	0.1	0.1	GC, GCMS
61.	1638	2149	α-Murrolol	0.5	0.6	GC, GCMS
62.	1669	-	khusinol	0.2	0.3	GC, GCMS

63.	1696	2313	(2*Z*,6*E*)-Farnesol	0.3	0.3	GC, GCMS
64.	1707	2330	**(Z,Z)-Farnesol**	**5.4**	**6.6**	GC, GCMS, CoGC
65.	1727	2347	(*E*,*E*)-Farnesol	0.5	1.0	GC, GCMS
66.	1796	-	Nootkatone	0.08	0.2	GC, GCMS
67.	1951	-	*epi*-13-Manool	0.06	0.8	GC, GCMS
68.	1976	2243	**Manoyl oxide**	**4.1**	**11.7**	GC, GCMS
69.	1994	-	*epi*-13-Manoyl oxide	0.3	0.5	GC, GCMS
70.	2038	-	**Abietatrine**	**1.1**	**3.7**	GC, GCMS
71.	2065	-	**Abietadiene**	**2.1**	**4.8**	GC, GCMS
72.	2242	-	*dehydro* Abietal	0.1	0.9	GC, GCMS
73.	2281	-	Abietal	0.6	2.1	GC, GCMS
74.	2330	-	4-*epi*-Abietol	0.2	2.3	GC, GCMS
			Yield (w/w)	0.26%	0.14%	
			Total oil	**93.35%**	**84.06%**	
			Grouped components			
			Hydrocarbon monoterpenes	**52.3%**	**38.26%**	
			Oxygenated monoterpenes	**23.19%**	**6.32%**	
			Hydrocaron sesquiterpenes	**1.32%**	**1.88%**	
			Oxygenated sesquiterpenes	**7.7%**	**12.2%**	
			Diterpene	**8.6%**	**21.1%**	
			Others	**0.3%**	**-**	

[a] Retention indices as determined on a DB-5 column using the homologous series of *n*- alkanes.
[b] Retention indices as determined on a HP-20M column using the homologous series of *n*- alkanes.
[c] Compounds listed in order of elution from a DB-5 column.,[d] Relative area was given according to FID area percentage data, [e] Trace (<0.05%), [f] Co GC = identification was based on retention times of authentic compounds on DB-5 capillary column.

Table2. The three most dominant components of the essential oils needles of several *J. communis*, as reported in the literature

Sample status	Origin	Year of publication	Three most important monoterpenes (max %)	Ref.
Dry	Greece	1993	α-Pinene (41.3%) Sabinene (17.4%) Limonene (4.2%)	[14]
Dry	Italy	1995	Sabinene (41.4%) α-Pinene (13.4%) Terpinen-4-ol (8.7%)	[15]
Dry	France	1997	α-Pinene (44.3%) Limonene (18.0%) β-pinene (11.1%)	[16]
Fresh	Sweden	1998	α-Pinene (56.8%) Limonene (6.9%) β-Phellandrene (6.9%)	[17]
Dry	Croatia	2000	α-Pinene (16.9%) Sabinene (12.1%) Terpinen-4-ol (7.7%)	[18]
Dry	India	2000	Sabinene (48.8%) α-Pinene (6.2%) *endo*-Fenchyl acetate (5.8%)	[27]

No specified	Italy	2003	Sabinene (61.1%) α-Pinene (6.4%) Terpinen-4-ol (10.7%)	[13]
Fresh	North Iran	2003	Sabinene (40.7%) α-Pinene (12.5%) Terpinen-4-ol (12.3%)	[19]
Dry	Iran	2004	δ-3-carene (39.4%) β-Phellandrene (25.1%) α-Pinene (24.5%)	[20]
Fresh	North-East Lithuania	2006	α-Pinene (66.5%) β-Phellandrene (12.5%) δ-3-Carene (5.2%)	[21]
Fresh	Vilnius District-Lithuania	2006	α-pinene (54.6%) β-Phellandrene (9.1%) Myrcene (2.5%)	[22]
Dry	Estonia	2010	α-Pinene (45.6%) Sabinene (15.4%) Limonene (4.6%)	[24]
Dry	Morocco	2011	Sabinene (27.5%) Limonene (16.2%) α-Pinene (8.2%)	[28]
No specified	China	2011	α-Pinene (27.0%) α-Terpineol (14.0%) Linalool (10.9%)	[25]
Fresh	Bulgaria	2013	α-Pinene (26.7%) Sabinene (19.6%) Terpinen-4-ol (5.2%)	[26]
Dry	Algeria	2014	α-Pinene (14.2%) Terpinen-4-ol (14.1%) Sabinene (12.4%)	This work

Conclusion

The essential oils of dried *J. communis* needles extracted by HD and MWHD, reported here for the first time, were mainly characterized by α-pinene, sabinene, terpinene 4-ol, γ-terpinene, (*Z*,*Z*)-farnesol and manoyl oxide. However, α-pinene, sabinene and terpinene 4-ol were detected in the essential oils of *J. communis* from Italy, Croatia, Northern Iran and Bulgaria, as main components, while δ-3-carene was reported only from the essential oil of *J. communis* collected from Iran.

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