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Chemical composition and stability of the hydrosols obtained during essential oil production. II. The case of *Picea glauca* (Moench) Voss., *Solidago puberula* Nutt., and *Mentha piperita* L.

François-Xavier Garneau, Guy Collin and Hélène Gagnon

Abstract
The aim of this work is to know if the hydrosols produced on a commercial basis are stable over at least 12 months at room temperature. The chemical composition of the oils and hydrosols collected from wild plantations were determined by GC-FID and GC/MS analyses. The components obtained from the hydrosols are mainly monoterpene alcohols, aldehydes and ketones and as such the composition of the hydrosols is different or very different from that of the corresponding oils: the oil with the most important fraction of the hydrocarbon compounds, the composition of the two phases is different. Benzyl alcohol, a compound not observed in the oil of *S. puberula* is by far the main compound (*ca*. 73%) observed in the corresponding hydrosol. Camphor is the main product observed in both oils (*ca.* 20%) and hydrosol (*ca.* 67%) of *P. glauca*. Menthol and menthone and their isomers (*ca*. 75%) are both observed in the oil and the hydrosol of *M. piperita*. These main compounds seem relatively stable in acidic water over the observation period.

Keywords: *Picea glauca, Solidago puberula, Mentha piperita*, hydrosol, shelf-life, essential oil, menthol, benzyl alcohol, camphor, p-mentha-1 (7), 8-dien-2-ol, p-menthadien-2-ol.

1. Introduction
In the previous paper of this series, we compared the composition of the essential oils and the fresh hydrosols obtained from two different species collected or cultivated in the Province of Quebec [1]. It was observed that the composition of the hydrosols changes slowly but significantly at room temperature. Thus, in the case of the hydrosols of *Melissa officinalis* L. and *Asarum canadense* L., a complex equilibrium involving nerol, geraniol, linalool, linalool oxides and several diols is not achieved after a two year shelf-life at normal temperature. The purpose of this work is to extend it to quantitatively important by-product of the essential oil production: the hydrosols of *Piceaglauca* (Moench) Voss., *Solidago puberula* Nutt., and *Mentha piperita* L. The aim of this work is to know if this chemical instability constitutes a general rule or if it must be considered as a case-to-case behaviour.

2. Materials and methods
2.1 Plant material
Aerial parts of *M. piperita*, Lamiaceae (Peppermint) and *S. Puberula*, Asteraceae (Downy goldenrod) were collected in October and in September, respectively, from spontaneous plants growing wild in the Grondines region, Quebec Province. The branches of *P. glauca*, Pinaceae (white spruce) were collected in October. The fresh material is cut into sections of two to four inches. All these materials are steam-distilled the day after the collection. These plants are very common in the Province of Quebec and in the North East part of North America. They are described in literature [2].

2.2 Oil and hydrosol extraction
All the samples were produced in a small plant located in Grondines, on the North side of the St-Lawrence River, between Quebec City and Three-Rivers. A typical batch involves 300 kg

of fresh material and produces 50 kg of hydrosol. A 4-liter bottle of each hydrosol was kept at room temperature during the time of experimentation. 100 mL hydrosols are submitted three times to extraction using 24 mL of chloroform HPLC grade solvent. These three fractions are gathered and are concentrated by partial evaporation until 2 mL and kept over dry $MgSO_4$. Liquid samples are stored in a room at −5 °C before the first GC or GC/MS analysis and then kept at room temperature for shelf stability studies.

2.3 Oil analysis

Fresh essential oils are analyzed by gas chromatography on an HP 5890, equipped with a flame ionization detector (GC-FID) and two capillary columns: a Supelcowax 10 and a DB-5 column (30 m × 0.25 mm × 0.25 µm). Samples are also analyzed by gas chromatography, HP 5890, coupled with an HP 5972 mass spectrometer at 70 eV(GC/MS), and equipped either with a DB-5 or a Supelcowax column (same as above). Injection port and detector temperature are 220 and 260 °C, respectively. The temperature program for both GC-FID and GC/MS is 40 °C for 2 min, then 2 °C/min until 210 °C and held under constant temperature for 33 min. Identification of the components is done by comparison of their retention indices (RI) with standards, by comparison of their mass spectra with literature data [3-5] and with our own databases. An internal standard, 400 µL (tetradecane: chloroform solution – 0.4:100) was added to the extract before each GC analysis. Quantitative data are obtained electronically from GC-FID area percentages. The FID response factors for compounds relative to tetradecane are taken as one.

2.4 Hydrosol analysis

They are submitted to GC and GC/MS analyses using the same procedure as that used for the essential oils. All the samples were tested for aerobic and facultative anaerobic heterotrophic bacteria to estimate the density of the bacterial population. The measured values are essentially 0, far below the accepted value for drinking water: <5 CFU/100 mL. Detection of coliforms or atypical bacteria is essentially negative in each case.

3. Results and discussion

3.1 *Piceaglauca*

The composition of the oil is well known [6-9]. The main oxygenated compounds of this oil are camphor and borneol. Several hydrocarbon monoterpenes such as camphene and limonene are also included in the oil (Table 1). The hydrosol was also described [10] and as such does not need a long description except for the behavior of the hydrosol during shelf-life studies. The present sample was analyzed four times. The fresh hydrosol contains 933 (± 6%) mg/L of volatile organic compounds (VOCs) of which 97% appear in Table 1. Due to the kinetic of extraction and particularly the quantity of water vapor used in the extraction process, the concentration of VOCs in hydrosol can change drastically. Moreover, the use of chloroform and the evaporation needed to obtain a sample suitable for GC analysis implies some uncertainties in analyzing quantities. pH value of each sample is 3.85 ± 0.05.

The six main compounds measured in the oil were α- and β-pinene (12.1 and 20.4%, respectively), camphene (7.7%), limonene (8.0%), camphor (20.3%), and bornyl acetate (11.8%). Hydrocarbon monoterpenes have a very low solubility in water at pH = 7: less than 5 mg/L. They are not observed in the acidic hydrosol. On the other hand, the camphor is a water soluble compound: *ca*. 1600 mg/liter [11]. Thus, it is the main compound observed in the hydrosol: > 65%. Several minor compounds, not observed in the oil, such as *trans*-3-hexenol (RI on DB-5: 859), tiglic acid (896), camphenilone (1091), α-campholenal (1136), nopinone (1141), *exo*-2-hydroxycineole acetate (1346), hydroxycitronellol (1362), vanillin (1393), methyl eugenol (1402), 4'-hydroxyacetophenone (1437), and acetovanillone (1479) in concentration lower than 0.1%, are not included in Table 1. Some of them are only detected by GC/MS.

The most important compound is camphor: about ⅔ of the VOCs extracted from the hydrosol. The variations in percentages for each compound are relatively of small importance. At a first glance, this percentage value is stable over a one-year conservation period at room temperature: from 65.5 to 68.1% (Table 1). About 90% of camphene hydrate disappears after one year. A careful look at the sum of the percentages of camphor + camphene hydrate seems quite constant: %(camphor) + %(camphene hydrate) = 68.25 ± 0.2 (Fig. 1A). The transformation of camphene hydrate into camphor is a possible route to explain this observation. This process would imply either the dehydration of camphene hydrate into camphene followed by an oxidation process to camphor, two processes relatively well-known, or an alternate mechanism. Further discussion of these possibilities is out of scope of this paper.

The explanations for the variations of the concentration of the other compounds are not obvious. Some percentages increase (*cis*-3-hexen-1-ol, *cis*-linalool oxide (fur.), borneol), some are rather constant (linalool, citronellol), and some decrease (1,8-cineole, terpinen-4-ol, piperitone, bornyl acetate). Linalool is linked with linalool oxides (fur.), nerol and geraniol as well as α-terpineol through isomerization/degradation processes [1]. Since the linalool percentage is very small, it does not play an important role in the acidic solution. It was shown that microbiological conversion of α-terpineol in the presence of *Pseudomonas aeruginosa* produced oleuropeic acid and borneol [12]. If this isomerization process is slowly working in acidic hydrosol free of this kind of bacteria, it may explain the variation in concentration observed in Fig. 1B.

Also, the relative stability of the sum, % (borneol) + % (bornyl acetate)= *ca*. 11.2%, at least during the 6 first months of storage, may be an indication of the hydrolysis of the acetate although such process would be quantitatively in contradiction with the above-mentioned possibility of the isomerization of α-terpineol towards borneol [12].

3.2 *Solidago Puberula*

The essential of *S. Puberula* was described elsewhere [13]. It has a very light blue color with refractive index: n_d = 1.4783. As it can be seen in Table 2, α-pinene (37%), α-pinene (6.9%), myrcene (11.1%), and limonene (11.9%) along with sesquiterpene germacrene D (12.4%) are the main compounds of the *S. Puberula* oil measured in our sample. The previously published analysis of this oil is different in that myrcene is the main product (53%) with α-farnesene as the main sesquiterpenes, 10.7% [13].

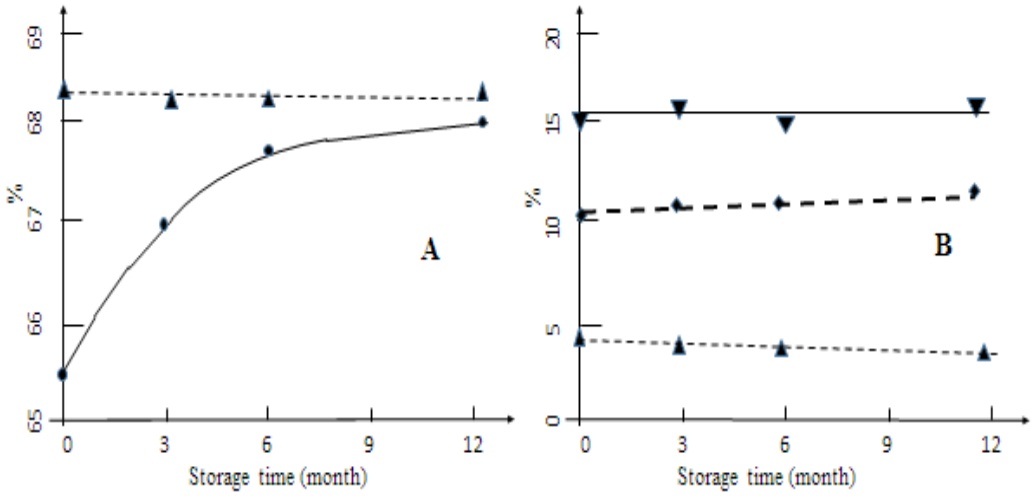

Fig 1: The variation in the composition of the hydrosol of *P. glauca*:
A: ●: camphor; ▲: camphor + camphene hydrate
B: ▲: α-terpineol; ♦; borneol; ▼; borneol + α-terpineol.

Table 1: Composition (%) of the hydrosol and partial composition of the essential oil of *Picea glauca*

	Shelf-life of hydrosol (month)		**0**	**3**	**6**	**12**	**Oil**
No	**Identification**	**RI** [1]					
1	furfural	835	0.1	0.1	0.1	0.1	-
2	*cis*-3-hexen-1-ol	863	2.4	2.6	2.7	2.7	-
3	hexanol	878	0.2	0.2	0.2	0.2	-
4	unidentified acid	939	0.1	0.1	0.1	0.1	-
5	hexanoic acid	1000	0.2	0.2	0.2	0.2	-
6	*trans*-3-hexenoic acid	1017	-	0.1	0.1	0.1	-
7	1,8-cineole	1035	2.0	1.9	2.0	1.7	1.0
8	benzyl alcohol	1047	t[2]	t	t	t	-
9	*trans-linalool oxide (fur.)*	1083	0.1	0.1	0.1	0.1	-
10	*cis-linalool oxide (fur.)*	1100	0.2	0.2	0.2	0.3	-
11	fenchone	1100	t	0.1	0.1	t	-
12	linalool	1115	0.3	0.3	0.2	0.2	0.2
13	exo-fenchol	1135	0.2	0.1	t	t	-
14	camphor	1154	65.5	67.0	67.6	68.1	20.2
15	camphene hydrate	1155	2.9	1.1	0.6	0.3	0.9
16	isoborneol	1162	0.2	0.3	0.4	0.4	-
17	borneol	1171	10.6	10.9	11.0	11.8	2.7
18	terpinen-4-ol	1180	1.5	1.4	1.4	1.1	0.4
19	*p*-cymen-8-ol	1185	1.1	1.0	0.9	1.1	0.1
20	α-terpineol	1191	4.4	4.0	3.9	3.5	0.9
21	*cis*-piperitol	1198	0.2	0.2	0.2	0.2	-
22	borneol isomer ?	1201	0.1	0.1	0.1	0.1	-
23	verbenone	1205	0.8	0.8	0.8	0.9	-
24	*trans*-carveol	1219	0.3	0.2	0.2	0.2	-
25	citronellol	1234	0.4	0.3	0.3	0.3	0.4
27	piperitone	1258	3.1	3.0	2.9	2.5	0.8
28	bornyl acetate	1294	0.6	0.3	0.2	0.2	-
29	unidentified A	1322	0.1	0.1	0.1	0.1	-
30	oplopanone	1736	0.3	0.2	0.2	0.3	-
	Total (%)		98	97	97	97	27.6

[1]: retention index on DB-5; [2]: traces, < 0.05 %; MS of unidentified compound A,*m/z*(intensity): 81(100), 43(58), 59(46), 96(28), 67(28), 41(16) … 153(12). (*p*-menthane-3,8-diol ?)

These differences may come either from the different locations of the plant as well as the time of collection or from phytochemical properties. Other significant mono and sesquiterpenes are camphene, α-ocimenes, α-caryophyllene, α-humulene and bicyclogermacrene. Except for bornyl acetate (2.0%), the other few oxygenated compounds have

concentration lesser than 0.2%. They are longipinanol, α-muurolol with traces of (*E*)-cinnamic acid and coumarin. The only common compounds to oil and hydrosol are linalool, borneol, terpinen-4-ol, bornyl acetate and α-cadinol (Table 3).

The fresh hydrosol has a pH of 4.1. This value decreases rapidly to 3.6 ± 0.1 during the three first months and stays constant during the next 9 months. As it is observed in the preceding paper [1], the measured compounds in the hydrosol are oxygenated. The total measured VOCs is 241 mg/L in the fresh hydrosol of which 96% are reported in Table 3. The total of VOCs is relatively stable during the experimental time. The low solubility of hydrocarbon compounds in pure water [8] explains their absence in fresh hydrosol.

On the other hand, the solubility of benzyl alcohol, the main compound observed in the hydrosol, is around 45 g/L in pure water at 25 °C [11]. In the extraction process, this compound is completely washed out from the oil as it is for several oxygenated compounds not observed in the oil. This is the case of furfuryl alcohol (RI on DB-5: 863), *sec*-phenylethyl alcohol (1069), acetophenone (1070), 6-camphenilone (1091), carvacrol (1310), eugenol (1360) and cinnamic acid (1435) (each less than 1 mg/L).

The main characteristic of the hydrosol are the presence of benzyl alcohol in high percentage: *ca*. 73 ± 1% and the relative stability of this value over storage time. This apparent stability may hide minor processes. The *ca*. 40% decrease in the hexanol percentage seems to be compensated by an increase in the hexanoic acid percentage: about 1.8%. The occurrence of various C_6 acids and the small increase of the concentration of hexanoic, *trans*-2-hexenoic and benzoic acid are also noticeable. This increase may explain part of the lowering of the pH values with shelf-time (Table 3). The complete vanishing of benzaldehyde could be related to the formation (at least in part) of benzoic acid. Thus, some oxidation processes seem to be at work. Obviously, because the way hydrosols are prepared, they contain molecular oxygen in solution.

As far as the behaviour of linalool is concerned, it was established that during the hops team distillation, linalool is transformed into isomeric furan and pyran linalool oxides, 2,6-dimethyl-7-octene-2,6-diol, and hodiendiol (2,6-dimethyl-3,7-octadiene-2,6-diol) [14]. The linalool oxides (pyr.) are measured at the traces level. 2,6-dimethyl-7-octene-2,6-diol is positively identified. Hodiendiol is not identified in the present study. The almost complete decrease of the percentage of linalool is not completely compensated by the increase of the other mentioned compound.

A question stays unanswered: are the oxygenated compounds extracted from the plant and present in the fresh hydrosol or are they formed during the distillation process?

3.3 *Mentha piperita*

Mentha piperita oil is well known and does not need to be described in details [7]. Menthone and menthol and their isomers are the main constituents of the oil. These compounds are present in the hydrosol (Table 4). The main hydrocarbon monoterpenes (*ca*. 3%) -among them limonene (1.5%) - and sesquiterpenes (*ca*. 2%) - germacrene D (0.9%) - are not observed in the hydrosol.

Table 2: Composition (%) of essential oil of *Solidago puberula*

Identification	RI[1]	RI[2]	This study	[13]
α-thujene	935	1028	t [3]	-
α-pinene	940	1020	37.2	9.4
camphene	953	1066	1.9	0.2
sabinene	976	1124	0.8	0.2
β-pinene	977	1108	6.9	1.0
myrcene	992	1170	11.1	53.0
α-phellandrene	1001	1165	0.6	5.7
p-cymene	1026	1277	0.1	0.1
limonene	1032	1195	11.9	2.8
β-phellandrene	1032	1202	0.5	-
cis-β-ocimene	1046	1244	t	0.6
trans-β-ocimene	1058	1262	1.5	6.0
terpinolene	1094	1289	0.1	-
linalool	1113	1557	0.1	-
borneol	1169	1698	0.1	-
terpinen-4-ol	1179	1594	0.1	-
bornyl acetate	1293	1574	2.0	0.5
α-copaene	1376	1489	0.2	-
β-cubebene	1389	1537	0.5	-
β-elemene	1391	1583	0.5	-
α-gurjunene	1405	1525	0.1	-
β-caryophyllene	1416	1583	2.8	1.2
trans-α-bergamotene	1437	1580	0.5	0.2
α-humulene	1454	1657	2.5	0.2
(*E*)- β-farnesene	1462	1670	-	2.0
α-amorphene	1483	1689	-	0.9
germacrene D	1483	1698	12.4	0.8
γ-amorphene	1494	1752	0.1	-
bicyclogermacrene	1498	1723	1.6	0.2
zingiberene	1501	1729	-	1.7
α-muurolene	1505	1719	0.2	0.1
germacrene A ?	1508	1749	0.6	-
α-farnesene	1514	1749	-	10.7
γ-cadinene	1517	1749	0.3	-
δ-cadinene	1527	1749	1.0	-
trans-nerolidol	1564	2040	0.1	0.5
longipinanol	1598		0.1	-
τ-muurolol	1640	2171	0.1	-
α-cadinol	1654	2213	0.2	-
α-bisabolol	1684	2201	-	0.1
Total			98.9	96.1

[1]: retention index on DB-5; [2]: retention index on Swax-10; [3]:traces <0.05%.

The fresh hydrosol contains 235 mg/L of VOCs of which 95% are reported in Table 3. pH values stay around 5.2 ± 0.2. The main characteristic of the hydrosol, in comparison with that of others hydrosols, is its relative stability over a one-year storage at room temperature. This is true for the menthols isomers: ca. 74%. Some minor compounds show an increase in their percentage: *cis*-3-hexen-1-ol, camphor… Others show a reverse behaviour. This is the case for menthone, pulegone, etc. Noteworthy is the vanishing of *p*-anisaldehyde after 3 months and the two oxygenated sesquiterpenols after a one year period of shelf storage. To our knowledge, unless it is formed during the distillation process, this is the first mention of 4-hydroxymenthol identified as a natural product. The MS of 4-neoisomenthol is available in literature [15] and the only

indication of the corresponding RI values taken in isotherm conditions are in line with our values [16]. The presence of three mintlactone derivatives not observed in the oil must be noticed. (-)-Mintlactone and (+)-isomintlactone were identified elsewhere as minor compounds in peppermint oil: 0.03 and 0.003%, respectively [17, 18]. 2,3-Didehydromintlactone is a rather rare compound. To our knowledge it was only identified in diethyl ether extract of woody material from *Burse ragraveolens* [19, 20].

The great similarity in the composition of the oil and of the hydrosol of *M. piperita* was recently recognized [21]. Menthol and menthone were identified as the main components in both phases. However, first, terpinen-4-ol and piperitone were identified only in the hydrosol and second, neomenthol and menthyl acetate were identified only in the oil. In the present study these products are observed in both phases. Finally, in agreement with our observations and as a consequence of their low solubility in aqueous media [22], germacrene D and limonene are only observed in the oil.

In the Inouye's paper [21], a classification of the hydrosols in two groups was proposed. Group A corresponds to hydrosols possessing major components common to that of oil. *M. piperita* was included in this group. Group B corresponds to hydrosols possessing major components different of those of the corresponding oil. Clearly, the hydrosol of *S. puberula* belongs to this group [1]. The hydrosol of *P. glauca* is rather in between these two groups.

Table 3: Composition (%) of the hydrosol of *S. puberula*

Identification	RI [1]	RI [2]	Storage time (month)				
			0	3	6	12	24
isoamyl alcohol	729	1208		0.2	0.4	0.4	0.3
n-pentanol	733	1208		0.1	0.3	0.3	0.2
furfural	835	1449	0.5	-	-	-	-
2-methylbutanoic acid	858		-	0.5	0.7	0. 7	0.6
trans-3-hexen-1-ol	859	1370	0.2	t [3]	t	t	t
cis-3-hexen-1-ol	863	1386	7.3	6.4	8.3	8.5	7.8
trans-2-hexen-1-ol	875	1407	0.7	0.4	0.4	0.3	0.2
hexanol	878	1361	1.5	1.0	1.0	0.9	0.5
benzaldehyde	962	1636	0.75	-	-	-	-
trans-3-hexenoic acid	987		-		0.4		t
hexanoic acid	998	1847	0.3	0.7	0.9	1.0	1.3
trans-2-hexenoic acid	1020	1967	-	0.4	0.5	0.5	0.7
benzyl alcohol	1047	1871	73.2	72.7	73.7	73.8	73.3
trans-linalool oxide (fur.)	1083	1442	0.3	0.3	0.3	0.3	0.3
cis-linalool oxide (fur.)	1100	1473	0.2	0.3	0.3	0.3	0.1
linalool (0.1%)*	1115	1557	3.2	2.3	1.6	0.7	0.1
hotrienol	1117		0.5	0.3	0.1	-	-
2-phenylethyl alcohol	1124	1905	0.3	0.7	0.6	0.6	0.6
borneol (0.1%)*	1171	1697	0.8	0.9	0.8	0.7	0.9
benzoic acid	1177	2382	-	1.0	1.1	1.3	1.3
terpinen-4-ol (0.1%)*	1180	1594	0.7	0.9	0.8	0.6	0.4
p-cymen-8-ol	1185	1846	0.4	0.4	0.3	0.4	0.4
α-terpineol	1191	1697	2.5	2.9	2.7	2.5	1.6
verbenone	1205	1691	0.2	0.3	0.2	0.2	0.2
trans-carveol	1219	1830	0.4	03	0.2	0.1	tr.
2,3-dihydrobenzofuran	1223	2352	1.5	1.4	0.9	0.5	0.1
nerol	1233	1796	0.3	0.2	0.1	tr.	tr.
citronellol	1233	1764	t	t	t	t	0.1
2,6-dimethyl-7-octene-2,6-diol	1233	1980	-	0.3	0.4	0.8	1.1
benzeneacetic acid	1259		-		0.3	0.3	-
geraniol	1263	1849	1.0	0.7	0.3	0.1	t
bornyl acetate (2.0%)*	1294	1573	0.2	t	t	t	-
terpin hydrate	1310	2090	-	0.2	0.2	0.6	1.2
cis-jasmone	1392	1931	0.2	0.1	0.1	0.1	0.1
α-cadinol (0.16%)*	1654	2213	0.3	0.2	0.1	t	-
Total (%)			96.5	96.0	97.6	96.8	96.1
Total (mg/L)			235	201	205	206	206

[1]: retention index on DB-5; [2]: retention index on Swax-10; [3]: traces (<0.1%); *: common compounds in oil and hydrosol (% in oil).

Table 4: Composition (%) of the hydrosol and of the essential oil of *M. piperita*

Shelf-life (month)			0	3	6	12	In oil
Identification	**RI [1]**	**RI [2]**					
cis-3-hexen-1-ol	863	1387	1.5	5.6	3.7	4.2	0.5_5
trans-2-hexen-1-ol	876	1407	t [3]	t	t	t	-
3-octanol	997	1390	0.2	0.2	0.2	0.2	-
1,8-cineole	1035	1201	0.4	3.5	1.2	1.4	3.7
benzyl alcohol	1047	1873	0.3	0.4	0.4	0.6	-
linalool	1114	1557	0.3	0.3	0.3	0.3	-
2-phenylethyl alcohol	1123	1905	-	-	-	0.3	-
cis-p-menth-2-en-1-ol	1131	1552	0.2	0.2	t	0.2	-
trans-p-menth-2-en-1-ol	1146	1646	0.3	0.3	0.4	-	-
camphor	1154	1507	0.9	1.6	1.4	1.6	-
menthone	1155	1459	5.5	4.5	3.0	2.4	15.9
isomenthone	1162	1488	1.5	1,8	1.6	1.5	2.0
borneol	1170	1691	t	t	t	t	-
neomenthol	1171	1592	3.3	2.7	2.8	2.6	4.0
δ-terpineol	1171	1667	0.5	0.5	0.6	0.6	-
menthol	1179	1640	69.4	61.7	69.3	68.8	52.3
terpinen-4-ol	1180	1594	2.7	3.0	3.3	3.5	0.8
iso-menthol	1185	1624	1.1	1.0	1.0	0.9	1.2
neo-iso-menthol	1189	1672	0.4	0.4	0.7	0.5	0.3
α-terpineol	1191	1698	0.6	0.6	0.4	0.7	-
2,3-dihydrobenzofuran	1223	2320	0.4	0.4	0.4	0.5	-
exo-2-hydroxy-1,8-cineole	1225	1830	t	t	t	t	-
pulegone	1241	1632	1.5	1.2	1.0	0.8	1.0
p-anisaldehyde	1255	1967	0.6	0.1	-	-	-
piperitone	1258	1716	1.8	1.8	1.9	2.0	0.4
unidentified A	1288		0.3	0.5	0.2	0.6	-
menthyl acetate	1294	1567	0.7	0.4	0.4	0.3	5.0
thymol	1302	2170	0.1	0.2	t	0.1	-
carvacrol	1312	2205	0.1	0.2	0.1	0.2	-
4-vinylguaiacol	1322	2167	1.0	0.4	0.3	-	-
4-hydroxymenthol (isomer of)[4]	1337	2081	t	t	t	t	-
trans-jasmone?	1388	1905	0.3	0.1	0.4	0.2	-
2,3-didehydromintlactone [5]	1493	2220	0.4	0.3	0.3	0.4	-
mintlactone	1496	2271	0.8	0.9	0.9	1.1	-
isomintlactone	1508	2306	0.2	0.2	t	0.1	-
globulol	1576	2033	0.3	0.1	0.1	0.0	0.3
α-cadinol	1654	2188	0.3	0.2	0.1	0.0	-
Total			98.5	94.8	96.3	96.3	87.6

[1]: retention index on DB-5; [2]: retention index on Swax-10; [3]: traces, <0.2%; [4]: tentatively identified[15, 16]; [5]: tentatively identified [17]; Mass spectrum of unidentified compound A, *m/z*(intensity): 119(100), 91(98), 79(95), 93(68), 84(68), 41(70), 93(66), 77(64), 55(45), 134(42), 92(38)

4. Conclusion

The composition of hydrosols obtained in the vapor distillation of *Piceaglauca*, is characterized by the presence of camphor in high percentage. The same situation appears in the case of *Solidago puberula* with benzyl alcohol. Menthols and menthones are also the main compounds observed in the hydrosol of *Mentha piperita*. These compounds seem very stable over at least a one year shelf-life. Thus, the composition of the hydrosols does not change significantly at room temperature.

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