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Accelerated autoxidation of essential oils: A study of oxidation resistance and protective effect of blends

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Abstract
In recent years, consumers have developed an ever-increasing interest in natural products as alternatives for every part of their lives. Among them, essential oils (EOs) used for aromatherapy had gained great interest regarding beauty, wellness and health. Constituted of wide and numerous ranges of volatile components, EOs are known to be susceptible to degradation reactions, such as oxidation. Changes in oil compositions may result in a loss of their properties or even in a new toxicity. Most of all, oxidated oils, as stronger allergens than native oils, are sources of great concern. Therefore, the present study aims to provide new data on the stability of Eos and blend of oils when exposed to air, a crucial parameter regarding EOs stability. Finally, the peroxide value analytical method was used as a parameter to follow-up the stability and oxidation rate of samples and, in the end, highlight potential protective effect of blends from oxidation.
The results show that most of the blends in the present study were oxidation-proof, unlike the tested Eos contained which were sensitive to oxidation when they were not part of a blend (e.g., sweet orange, lemon and eucalyptus EOs).
In conclusion, the study shows an improvement of essential oil stability by blending, which has never been described in the past, and which shows a real interest of blending essential oils.

Keywords: Essential oils (EOs), antioxidant, peroxide value, stability

1. Introduction
Essential oils (EOs) are liquid extracts commonly obtained from steam distillation of plant materials. As concentrates of odorant molecules, EOs are frequently used as a fragrance ingredient in perfumes and cosmetics. They had also been used for centuries in traditional herbal medicine and nowadays in well-documented alternative and complementary medicine such as aromatherapy, based on antibacterial, antioxidant or relaxing properties [1]. EOs are complex mixtures of different compounds which vary depending on which species and what parts of the plant are distilled but also on the origin of the plant or the time of harvest.
Several compounds are known to readily oxidize when exposed to air, reacting with oxygen in presence by a process called autoxidation. The common mechanism involves the addition of oxygen to first form peroxides and more precisely hydroperoxides. In EOs, hydroperoxides resulting from oxidation have been identified and found to be stronger allergens than the native compounds [2] as well as stronger irritants [3]. For instance, native linalool and limonene are very weak sensitisers while the corresponding hydroperoxides are stronger, as documented both experimentally [4-5] and clinically [6-7]. Moreover, secondary oxidation products from terpenes have also shown some allergenic activity (e.g., allergenic epoxides identified in oxidized limonene) [5].
The aim of the present study was to investigate if blends can influence the autoxidation of EOs contained in them or, even better, protect them from oxidation. Blends of EOs and pure EOs were exposed separately to a forced air stream and the autoxidation was followed thanks to chemical analysis of peroxide values.

2. Materials and Methods
2.1 Samples
For the first step of the study, lavender EO (*Lavandula latifolia* Medik.– Appendix A1), sweet orange EO (*Citrus sinensis* (L.) Osbeck – Appendix A2), rosemary EO (*Rosmarinus officinalis* L. (ct. verbenone) – AppendixA3) and a blend of 50/50 (v/v) sweet orange EO / rosemary EO (ct. verbenone) were obtained from Omega Pharma (France).

For the second step of the study, blends of EOs and pure EOs were obtained from Florame (France) (n°1), from Pierre Fabre-Naturactive (France) (n°2-3) and from Puressentiel (France) (n°4). Detail of the samples and composition of the blends are summarized in Table 1.

Table 1: Samples of blends and associated EOs with chromatographies in appendix

N°	Blend compositions	Pure EOs	Appendix
1	Sweet orange, rosemary (ct. cineol), lemon, grapefruit	Sweet orange *(Citrus sinensis* (L.) Osbeck*)* Rosemary (*Rosmarinus officinalis* L. (ct. cineol))	A4 A5
2	Lemon, litsea, sweet orange, eucalyptus *globulus*, rosemary (ct. cineol), lavandin grosso, star anise, thyme, tea tree and Atlas cedar	Eucalyptus *globulus* (*Eucalyptus globulus* Labill.) Lemon (*Citrus limon* (L.) Osbeck)	A6 A7
3	Sweet orange, eucalyptus *globulus*, rosemary (ct. cineol), red myrtle, Scots pine and black spruce	Eucalyptus *globulus* (*Eucalyptus globulus* Labill.)	A6
4	Dill, anise, tea tree, exotic basil, St Thomas bay, camphor tree, cajeput, Chinese cinnamon, Atlas cedar, lemon, Java citronella, cumin, cypress, eucalyptus *globulus*, sweet fennel, gaultheria, juniper, rose geranium, fresh ginger, clove, true lavender, lavandin, lemongrass, mace, mandarin, marjoram, lemon balm, spearmint, peppermint, myrrh, niaouli, sweet orange, oregano, parsley, petit grain bigarade, Scotch pine, rosemary (ct. cineol), savory, sage, wild thyme and thyme	Clove (*Syzygium aromaticum* (L.) Merr. &L. M. Perry) Mandarin (*Citrus reticulata* Blanco)	A8 A9

2.2 Analysis of peroxide value

The peroxide value (POV) determines the degree of oxidation in EOs. The analytical method involves the reaction of the hydroperoxides with potassium iodide added as a reducing agent, followed by the titration of iodine produced and a calculation to determine the number of peroxides in 1000g of sample. POV are expressed in milli-equivalents (mEq) of active oxygen per kilogram of sample. A high POV means a high amount of peroxide in the sample, corresponding to an advance state of oxidation.

5g of sample were dissolved in 30mL of a mix of chloroform / acetic acid (2/3) (v/v). This solution wasadded to 0.5mL of a saturated potassium iodide solution and agitated for exactly 1min. The reaction wasstopped by adding 30mL of purified water and 5mL of starch solution. The mixture was titrated with 0.1M sodium thiosulfate solution. The POV is calculated from the following formula:

$$POV = (V*M*1000)/W \quad (1)$$

Where V = volume of titrating solution (mL), M = molarity of the thiosulfate solution; W = weight of sample (g).

2.3 Air exposure and autoxidation follow-up

Samples of pure EOs and blends of EOs were placed on a magnetic plate, agitated and exposed to a bubbling air stream in open Erlenmeyer flasks (300mL for step 1, 450 mL for step 2). This procedure was used to accelerate the natural oxidation phenomenon. POVs were determined at the beginning and then samples were collected and analyzed on a regular basis until 40 days according to table 2.

Table 2: Sampling times for peroxide value analysis

Step	Sampling times
1	0h, 4h, 24h, 48h, then 5, 7, 9, 12, 15, 19, 22, 26, 29, 33, 26 and 40 days.
2	0, 5, 10, 15, 20, 25, 30, 35 and 40 days.

3. Results

3.1. Step 1

Under the conditions of the accelerated oxidation experience, initial values ranged from 1 (rosemaryct. Verbenone EO) to 8 (sweet orange EO) mEq of O_2/kg. POVs rose very slowly for rosemary ct. verbenone EO. The blend of sweet orange-rosemary (ct. verbenone) EO followed the same trend to reach 22 and 23 mEq of O_2/kg, respectively. POVs of lavender EO and sweet orange EO increased rapidly, reached a peak at very high values and then began to decline until all the EOs evaporated (figure 1). Sweet orange EO led to the highest POVs, with a peak of 322 and a terminal value of 265 mEq of O_2/kg,and had the fastest evaporation (only 22 days).

3.2 Step 2: pure EOs

Several EOs evaporated totally before the 40th day (after 20, 25 or 30 days) (figure 2). Under the conditions of the accelerated oxidation experience, the amounts of peroxide ranged from 3.8 (clove EO at day 0) to 200 (lemon EO at day 30) mEq of O_2/kg. The POV rose very slowly for clove EO, progressively and more quickly for eucalyptus and rosemary (ct. cineol) EOs, first slowly and then strongly after the 15th day for the citrus EOs: mandarin, orange and lemon. Only clove EO had low POV while others reached very high values.

3.3 Step 2: Blends and corresponding EOs

Under the conditions of the accelerated oxidation experience, the amounts of peroxide ranged from 2.8 (blend n°4 at day 0) to 105.9 (blend n°3 at day 30) mEq of O_2/kg. Blend n°1 and rosemary (ct. cineol) EO followed the same trend: high initial values rising moderately. Blend n°2 was almost stable during the 40 days. The pure EOs constituting the blend and tested separately (lemon and eucalyptus) did not follow the blend n°2 trend. Blend n°3 and eucalyptus EO had some similar values until the 20th day but they diverged after. Blend n°4 followed the same trend as clove EO, with low and almost stable POVs (figure 3).

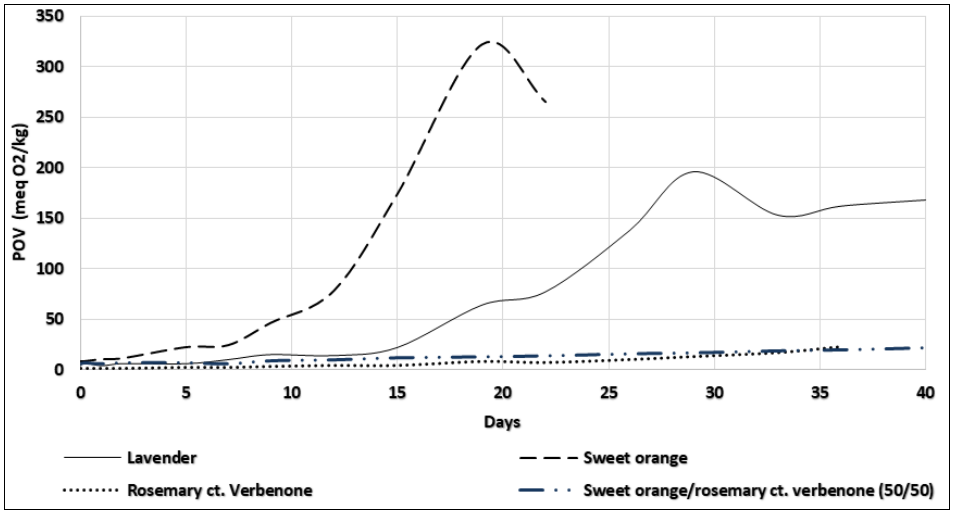

Fig 1: Step 1: peroxide values under accelerated autoxidation

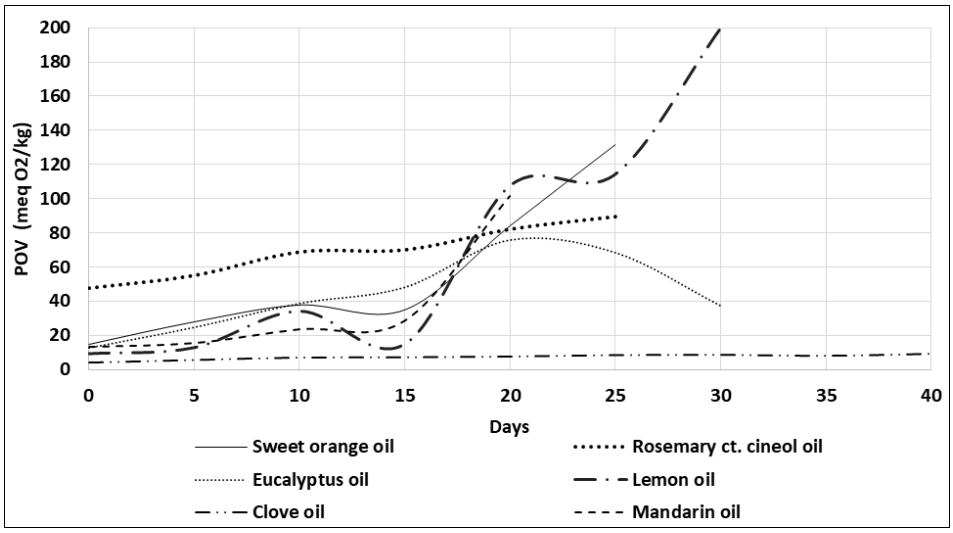

Fig 2: Step 2: peroxide values of pure EOs under accelerated autoxidation

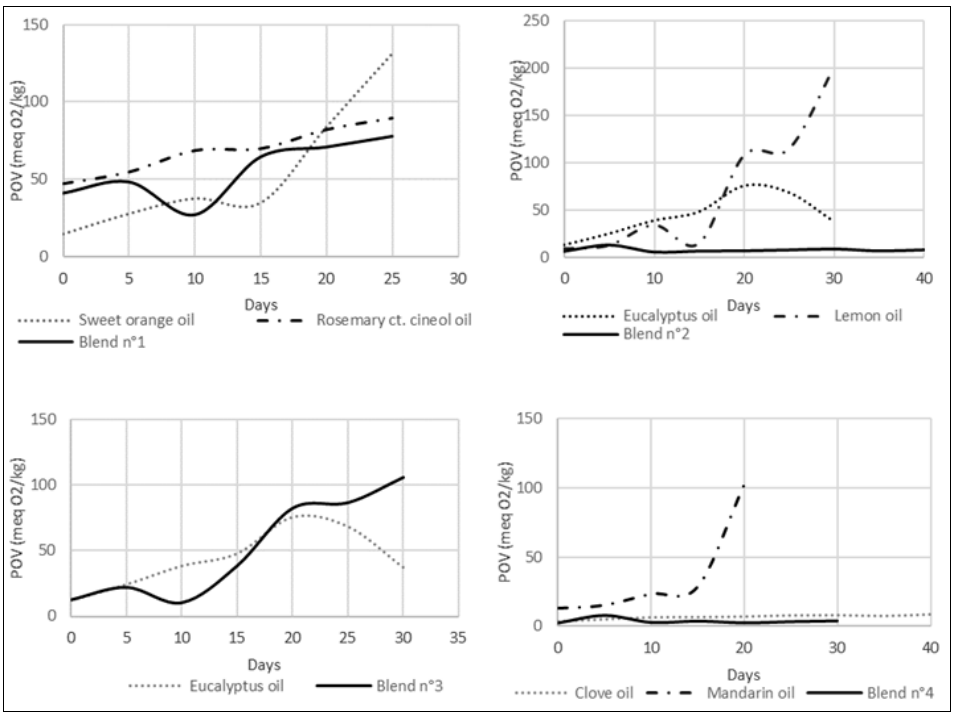

Fig 3: Pure oils and blends peroxide values follow-up under accelerated autoxidation parameters: blend n°1 (top left), n°2 (top right), n°3 (bottom left), n°4 (bottom right)

4. Discussion

In the present study, peroxide values of some pure EOs and blends of EOs were measured to evaluate and follow the state of oxidation under accelerating parameters. Blend evolutions were compared only with EOs provided by the same company, to prevent sourcing divergences. EOs are partly composed of molecules with unsaturated hydrocarbons such as terpenes (e.g., limonene and linalool as presented in figure 4) prone to oxidation if exposed to air, forming hydroperoxides which react further to form secondary oxidation products (e.g., alcohols, ketones, aldehydes or polymeric material). Meanwhile, EOs, as volatile substances, evaporate, leading to concomitant phenomena: oxidation, degradation and evaporation. After some point, when degradation becomes the dominating process, the concentration of peroxides decreases resulting in lower POV. This classic evolution was observed in step 1 with lavender EO and sweet orange EO and in step 2 with eucalyptus EO. With the other EOs we can assume that they evaporated before we can see the decrease.

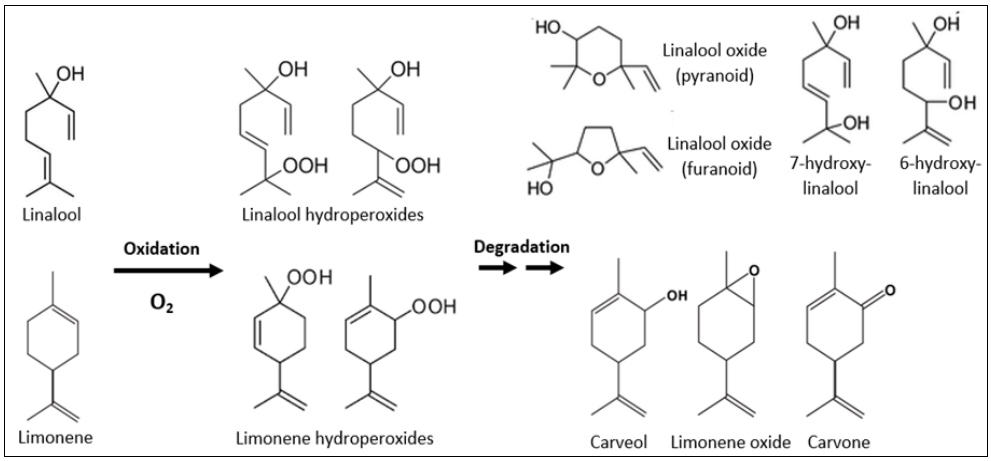

Fig 4: Main oxidation and degradtion products of linalool (main compound of lavender EO) and limonene (main compound of sweet orange, lemon and mandarin EOs)

In this study, during the first step, lavender and sweet orange EOs have shown to oxidize more and faster than rosemary (ct. verbenone) EO. The blend of sweet orange and rosemaryct. verbenone EOs was very stable suggesting a protective effect on the sweet orange EO which is usually very sensitive to oxidation when it is pure and not part of a blend. Likewise, during the second step, blend n°1 and rosemary (ct. cineol) EO following the same trend, we can assume that rosemary (ct. cineol)EO prevents the oxidation of the citrus EOs in blend n°1. Blend n°2 was very stable during the whole study: this blend is oxidation-proof unlike the tested EOs contained (lemon and eucalyptus EOs). This behavior should be due to one or several EOs or even a synergy of the other non-tested EOs among litsea, sweet orange, rosemary, lavandin grosso, star anise, thyme, tea tree and Atlas cedar. In contrast, blend n°3 was very prone to oxidize. The different EOs in the blend (sweet orange, eucalyptus *globulus*, rosemary (ct. cineol), red myrtle (*Myrtus communis* L.), Scots pine (*Pinus sylvestris*)) and black spruce (*Picea mariana*) and/or their concentrations did not exert a protective effect. Finally, blend n°4, as long as the clove EO it contained, were very stable during the whole study. The blend may have prevented the oxidation of the present EOs. However, containing 41 EOs, the blend was too complex to identify at this step which EO(s) may contribute to its stability.

We have shown the peroxides to be rapidly formed in really high amounts for almost all pure EOs. Only rosemary (ct. verbenone) EO (step 1) and clove EO (step 2) had very low and stable POVs compared with the other EOs. They seem to be protected from autoxidation and, at once, could be used as antioxidant to protect other EOs by strongly inhibiting the oxidation of unstable components in a blend. Phenolic compounds are the main direct antioxidants components, acting by formal hydrogen atom transfer, together with indirect antioxidant cyclohexadiene-like with a chain termination mechanism [8]. EO compositions can help roughly to predict an antioxidant potential for the ones with a good proportion of phenolic compounds (e.g., carvacrol, thymol or eugenol) [9] or cyclohexadiene derivatives (α and γ-terpinene, α-phellandrene), as shown in figure 5, and a modest proportion of unsaturated terpenes. Nevertheless, the overall antioxidant effect is, in fact, the result of the complex composition of each EO, with possible synergistic or antagonistic effects with each other [10].

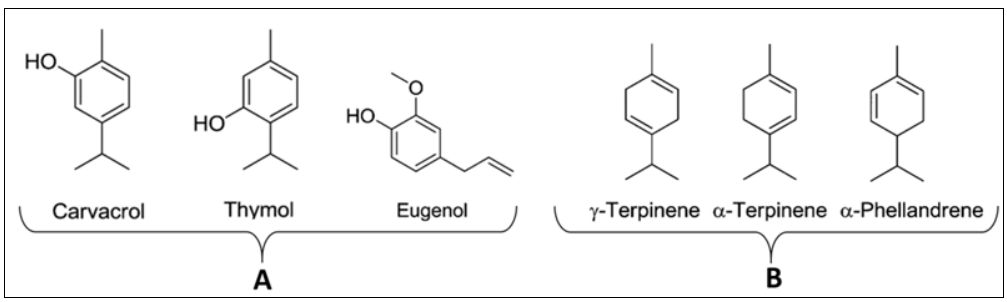

Fig 5: Common antioxidant components in essential oils. Group A: example of phenolic compounds. Group B: example of cyclohexadiene-like compounds

Different chemotypes of rosemary EO coexist with commonly a range of concentration in α-pinene, 1, 8-cineole, verbenone, borneol and camphor [11]. Non-phenolic nor cyclohexadiene-like components which could justify a protective antioxidant effect are presents. In the literature, some antioxidant tests achieved to be positives whereas the opposite conclusion can be reached depending on the testing method applied. Rosemary EO could be co-oxidized but also show an apparent antioxidant activity at the same time. As far, it is not possible to explain the antioxidant protective effect observed in our study. Clove EO, for its part, is almost 90% composed of eugenol, which, as a phenolic compound, is known to be a good antioxidant [12-14] confirming our study.

Oregano (*Origanum vulgare*) [15], thyme (*Thymus vulgaris*) [13], and black cumin (*Nigella sativa*) [16] had been tested as examplesof the best potential antioxidant EOs. Others, such as wild thyme (*Thymus polytrichus*) [15], winter savory (*Satureja montana*) [15], sage (*Salvia officinalis*) [13], sweet basil (*Ocimum basilicum*) [12, 14] or coriander (*Coriandrum sativum*) [17] have a much lower antioxidant activity. Nevertheless, when they are mix together, they can be present in really high concentrations and so it is likely that they can produce a significant or synergic antioxidant effect, as seen in blend n°4.

While this study, as a worst-case scenario, proved very useful to evaluate and compare the potential of different EOs to spontaneously oxidize (by autoxidation), it cannot be used and extrapolated to how EOs evolve in consumer products. Indeed, open flasks, agitation and a bubbling air stream were very adverse conditions while manufacturers recommend to store products well closed and away from light. Nevertheless, we noticed some samples were barely influenced by those parameters, suggesting an even better preservation under normal conditions of storage.

5. Conclusions

The study shows an improvement of essential oil stability by blending, which has never been described in the past, and which shows a real interest of blending essential oils.

Further studies should be carried out exposed to highlight which EOs and concentrations are useful to stabilize a blend and extend its preservation.

Abbreviations

ct. EO(s)	Chemotype Essential oil(s)
mEq	milli-equivalents
mL	milliliters
POV(s)	Peroxide value(s)
v/v	volume/volume

6. Acknowledgments

The technical assistance of Omega Pharma France's CQ lab is gratefully acknowledged, as much as Lexva Analytique lab, for POV analysis. We thank Florame, Pierre Fabre-Naturactive and Puressentiel for providing EOs samples.

7. References

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Abbreviations

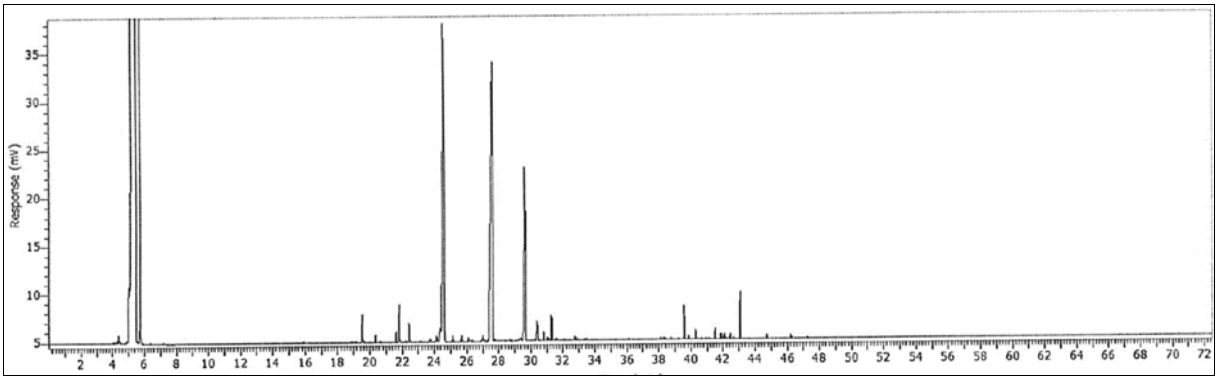
ct. EO(s)	Chemotype Essential oil(s)
mEq	milli-equivalents
mL	milliliters
POV(s)	Peroxide value(s)
v/v	volume/volume

Appendix

This appendix contains the chromatographic reports of pure EOs samples analyzed.

A1. Lavender EO (*Lavandula latifolia* Medik.)

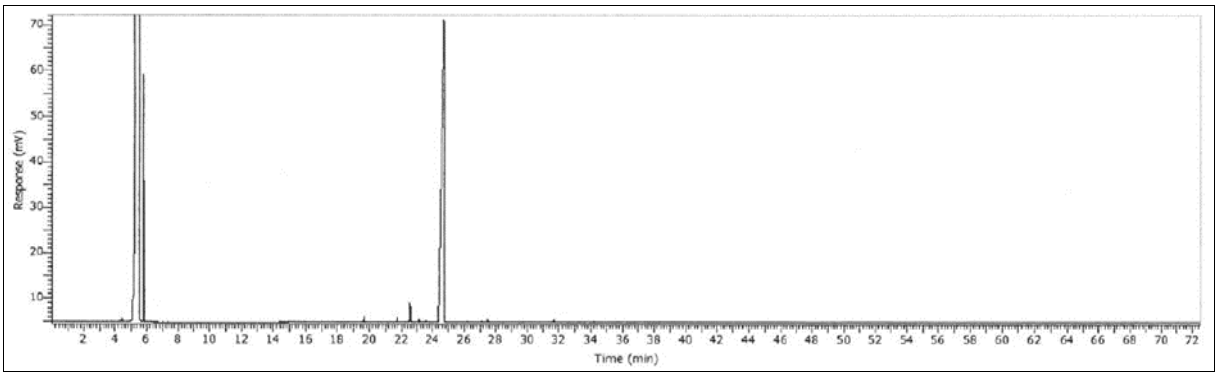
Instrument	GC Perkin Elmer Clarus 500	T° program (°C)	50°C (10') > 240°C (15') : 4°C/min (73')
Column	5ms 60m*0.25mm*0.25µm	Auto injection	1.0µL
Split injector	250°C	Sample dilution	3% in hexane
Vector gas	Helium 1.5 mL/min	Split	10 : 1



Caracteristic peaks	Results (%)	Caracteristic peaks	Results (%)
Limonene	1.3	Linalyl acetate	0.1
1,8-cineole	27.4	α-terpineol	1.0
Camphor	12.3	*trans*-α-bisabolene	2.0
Linalool	41.3		

A2. Sweet orange EO (*Citrus sinensis* (L.) Osbeck)

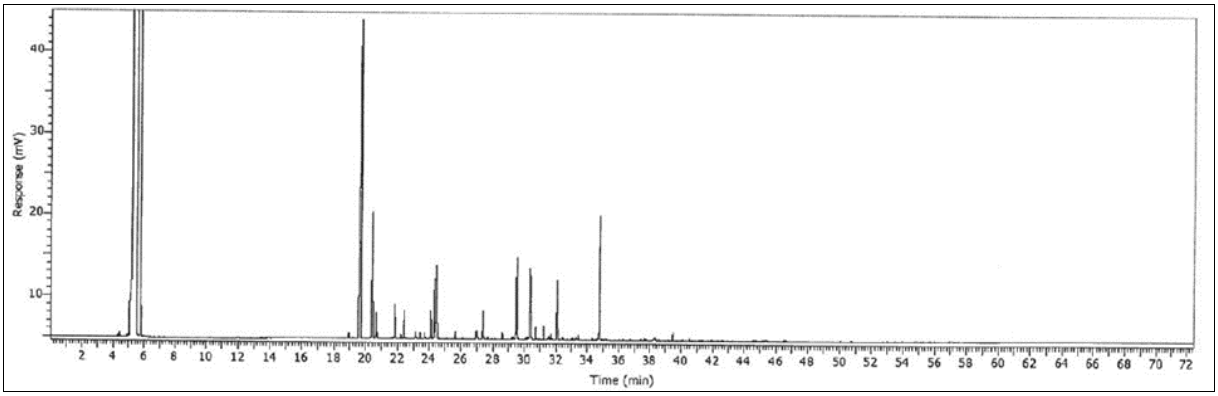
Instrument	GC Perkin Elmer Clarus 500	T° program (°C)	50°C (10') > 240°C (15') : 4°C/min (73')
Column	5ms 60m*0.25mm*0.25µm	Auto injection	1.0µL
Split injector	250°C	Sample dilution	3% in hexane
Vector gas	Helium 1.5 mL/min	Split	10 : 1



Caracteristic peaks	Results (%)	Caracteristic peaks	Results (%)
α-pinene	0.5	Linalool	0.2
Sabinene	0.4	Decanal	0.2
ß-pinene	0.04	Neral	0.04
ß-myrcene	1.7	Geranial	0.08
Octanal	0.3	Valencene	0.04
Limonene	95.4		

A3. Rosemary EO (*Rosmarinus officinalis* L. (ct. verbenone))

Instrument	GC Perkin Elmer Clarus 500	T° program (°C)	50°C (10') > 240°C (15') : 4°C/min (73')
Column	5ms 60m*0.25mm*0.25µm	Auto injection	1.0µL
Split injector	250°C	Sample dilution	3% in hexane
Vector gas	Helium 1.5 mL/min	Split	10 : 1



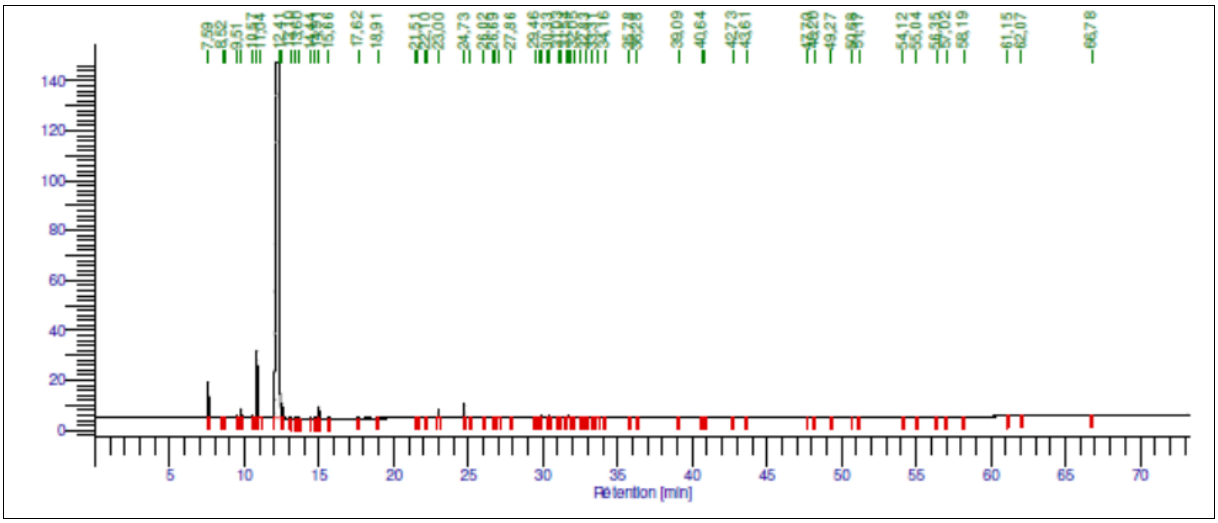
Caracteristic peaks	Results (%)	Caracteristic peaks	Results (%)
α-pinene	39.8	Linalool	1.6
Camphene	9.1	Camphor	5.6
ß-pinene	1.9	Borneol	5.8
Limonene	4.1	Verbenone	4.2
1,8-cineole	4.1	Bornyl acetate	8.7

A4. Sweet orange (*Citrus sinensis* (L.) Osbeck)

Instrument	GC-FID/FID
Polar column	Elite Vax (100% polyethyleneglycol) 60 m /0.25 mm/0.25 µm
Apolarcolumn	Elite 5 (5% diphenyl 95% dimethylpolysiloxane) 60 m /0.25 mm/0.25 µm
Vector gas	hydrogen

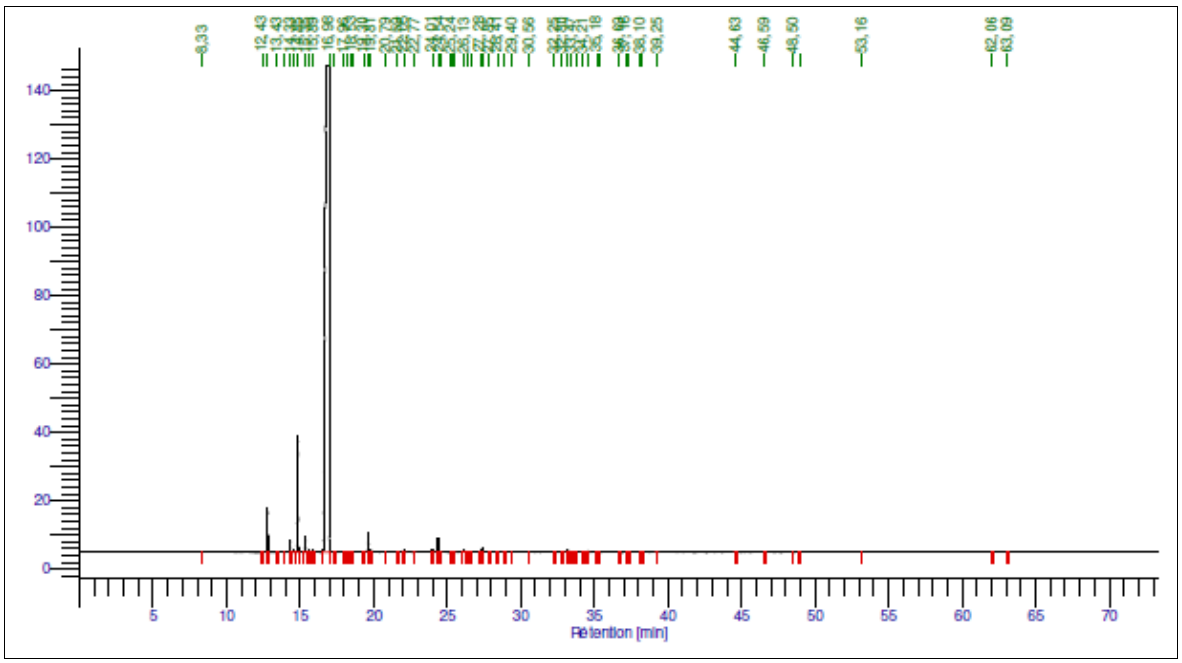
Polar column

Pics ordre	Molécules Noms	Retention [min]	Aires [uV*sec]	Aires [%]
1	ALPHA PINENE	7,592	35567,23	0,63
5	SABINENE	9,786	10769,97	0,19
6		10,569	3589,72	0,06
7	MYRCENE	10,852	105596,74	1,88
9	LIMONENE	12,408	5397667,30	96,16
10	G-TERPINENE	12,533	13693,67	0,24
18	OCTANAL	14,994	12909,84	0,23
26	N DECANAL	23,001	11527,19	0,21
27	LINALOL	24,726	17619,16	0,31
42	GERANIAL	31,695	4304,26	0,08
		158,157	5613245,08	100,00



Apolar column

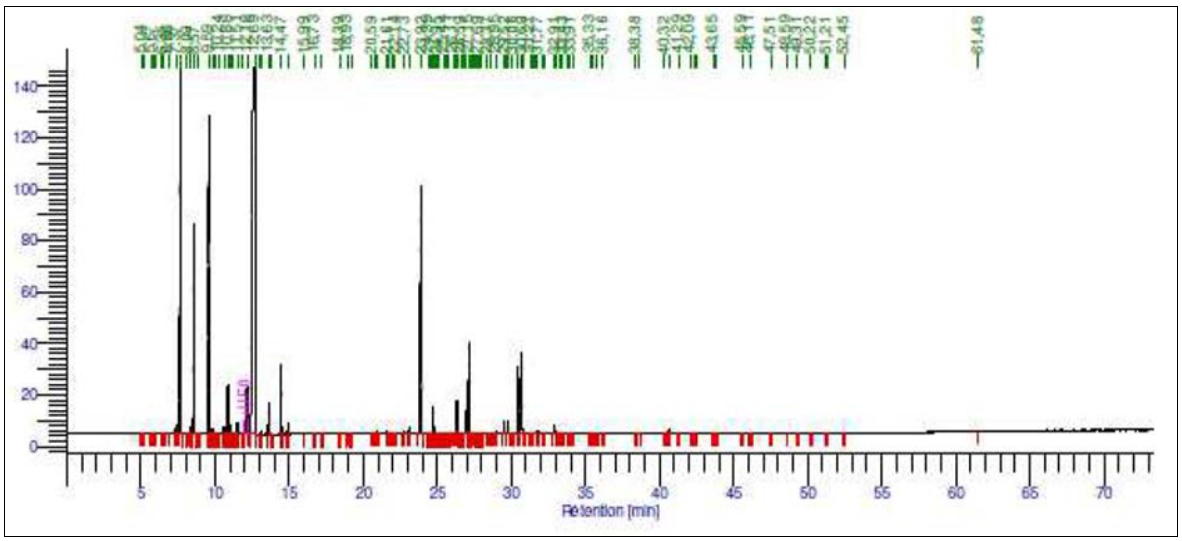
Pics ordre	Molécules Noms	Rétention [min]	Aires [uV*sec]	Aires [%]
3	ALPHA PINENE	12,790	35672,62	0,66
6	SABINENE	14,326	10825,62	0,20
8	MYRCENE	14,851	106402,25	1,97
9	N-OCTANAL	15,352	13961,94	0,26
11		15,885	3627,49	0,07
12	LIMONENE	16,983	5207266,69	96,23
19	LINALOL	19,638	17379,29	0,32
26	N-DECANAL	24,382	11751,30	0,22
37	GERANIAL	27,400	4168,08	0,08
		161,608	5411055,29	100,00



A5. Rosemary (*Rosmarinus officinalis* L. (ct. cineole))

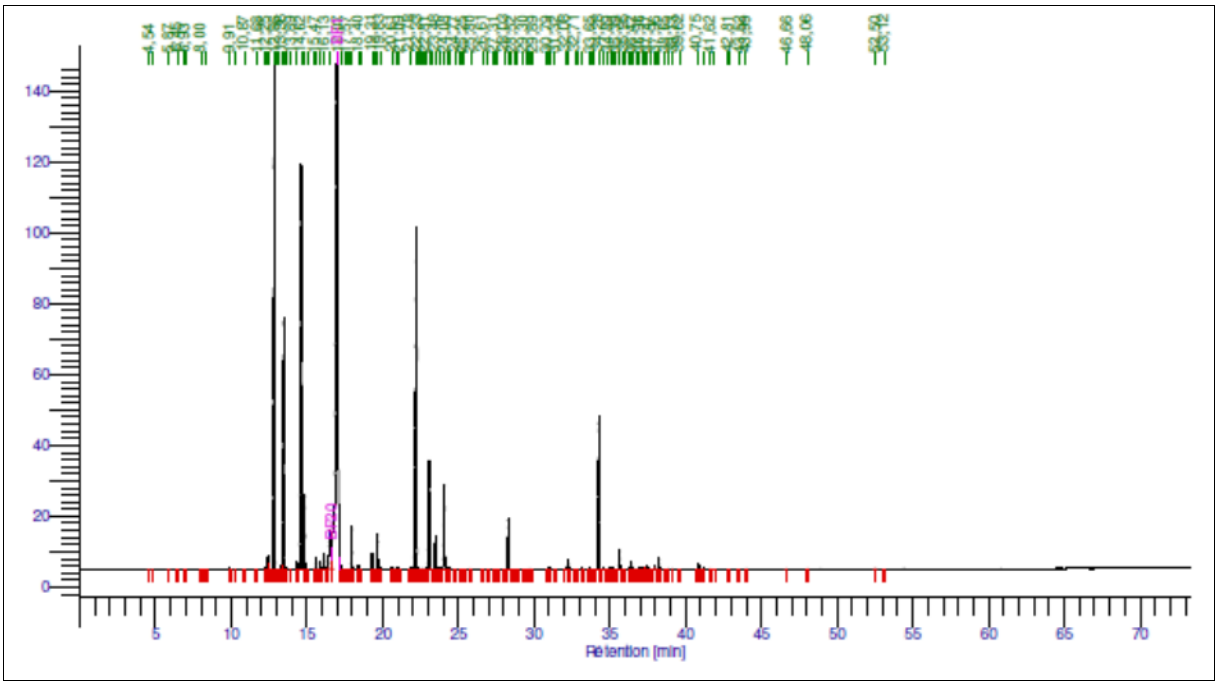
Instrument	GC-FID/FID
Polar column	Elite Vax (100% polyethyleneglycol) 60 m /0.25 mm/0.25 µm
Apolarcolumn	Elite 5 (5% diphenyl 95% dimethylpolysiloxane) 60 m /0.25 mm/0.25 µm
Vector gas	hydrogen

Polar column



Pics ordre	Molécules Noms	Rétention [min]	Aires [uV*sec]	Aires [%]
9		7,371	9011,38	0,17
10	alpha pinene+alpha thuyene	7,666	693487,50	13,24
12	ALPHA FENCHENE	8,364	5848,62	0,11
13	CAMPHENE	8,573	215339,26	4,11
15	B-PINENE	9,589	420432,67	8,03
16		9,819	5279,05	0,10
20		10,595	7400,51	0,14
21	Myrcene	10,879	58256,34	1,11
22	A-PHELLANDRENE	11,061	9229,72	0,18
24	A-TERPINENE	11,505	15410,27	0,29
26	LIMONENE	12,176	133680,62	2,55
27	1,8-cineole	12,689	2422889,32	46,26
30	G-TERPINENE	13,628	34812,73	0,66
33	P-CYMENE	14,471	80154,42	1,53
34	TERPINOLENE	14,933	12120,13	0,23
43		20,946	4058,47	0,08
44		21,614	4072,78	0,08
48		22,734	2928,44	0,06
49	ALPHA COPAENE	23,112	8581,99	0,16
50	CAMPHRE	23,917	547264,93	10,45
53	LINALOL	24,741	30359,90	0,58
61		26,220	3011,54	0,06
62	ACETATE DE BORNYLE	26,323	43984,09	0,84
65	TERPINEN -4-OL	26,969	30690,08	0,59
66	B-CARYOPHYLLENE	27,147	162064,56	3,09
76		28,950	2587,46	0,05
77		29,518	16974,33	0,32
79	alpha caryophyllene	29,767	18248,44	0,35
82	A-TERPINEOL	30,473	94481,33	1,80
83	BORNEOL	30,692	122068,89	2,33
89		31,773	3721,56	0,07
92	verbénone	32,912	10114,20	0,19
93		33,014	4175,43	0,08
106		40,632	5359,42	0,10
		694,777	5238100,37	100,00

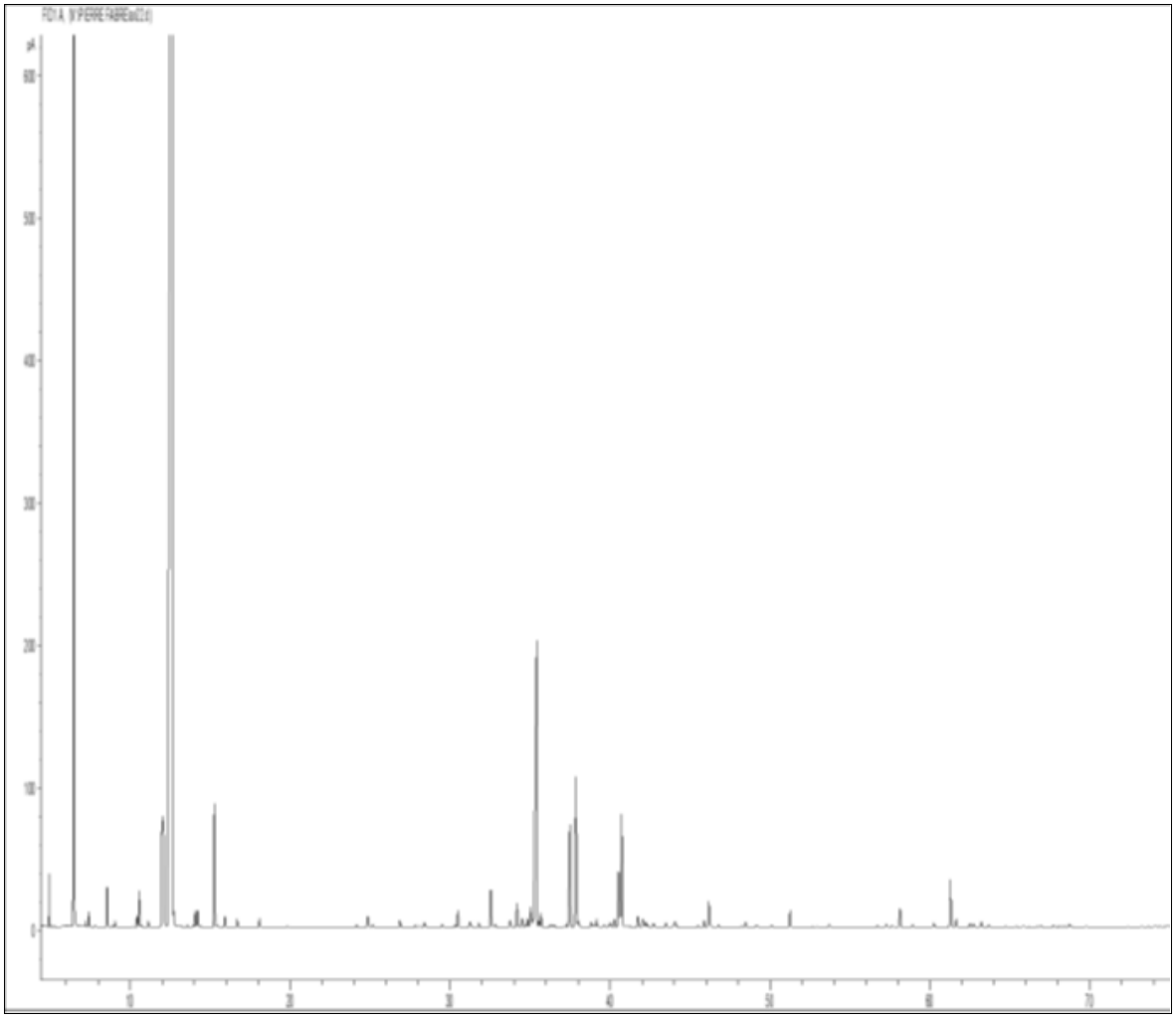
Apolar column



Pics ordre	Molécules Noms	Rétention [min]	Aires [uV*sec]	Aires [%]
15		12,391	9086,10	0,17
16	A-THUYENE	12,453	10970,19	0,21
17	ALPHA PINENE	12,864	669109,36	12,83
20	ALPHA FENCHENE	13,363	5675,56	0,11
21	CAMPHENE	13,471	216281,83	4,15
24	SABINENE	14,351	9665,18	0,19
25	B-PINENE	14,624	418789,20	8,03
26	MYRCENE	14,858	59960,16	1,15
29	ALPHA PHELLANDRENE	15,615	11109,72	0,21
30		15,893	7413,63	0,14
31	A-TERPINENE	16,135	15867,98	0,30
32	P-CYMENE	16,554	81307,82	1,56
33	1.8 CINEOLE + LIMONENE	17,072	2523183,90	48,39
38	G-TERPINENE	17,966	35930,65	0,69
39		18,403	3955,67	0,08
41	TERPINOLENE	19,311	14444,36	0,28
43	LINALOL	19,668	31546,42	0,60
51	CAMPHRE	22,238	550688,63	10,56
57	BORNEOL	23,080	140689,99	2,70
59	TERPINEN-4-OL	23,485	30688,57	0,59
61	A-TERPINEOL	24,079	88863,44	1,70
76	ACETATE DE BORNYLE	28,292	46032,26	0,88
89		32,078	2522,24	0,05
90		32,270	8694,66	0,17
97	TRANS-B-CARYOPHYLLENE	34,280	167255,70	3,21
100		35,030	2604,05	0,05
103	A-CARYOPHYLLENE	35,636	18293,50	0,35
107		36,415	8034,15	0,15
113		37,334	2633,49	0,05
114		37,472	2722,18	0,05
116		37,959	4209,70	0,08
118		38,236	10866,43	0,21
124		40,868	5486,98	0,11
		783,744	5214583,69	100,00

A6. Eucalyptus *globulus* (*Eucalyptus globulus* Labill.)

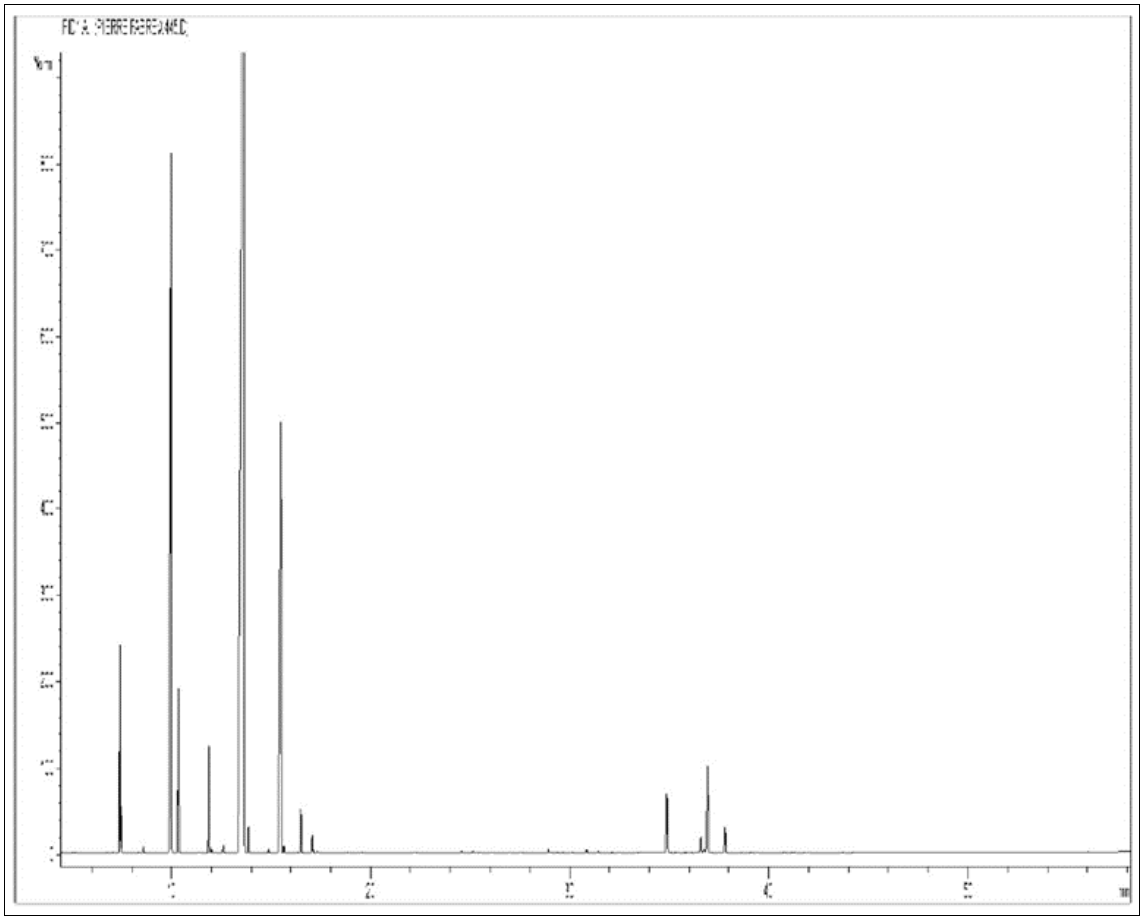
Instrument	**GC/FID Agilent 6890**	**T° program (°C)**	**60°C (5') > 250°C (15') : 2°C/min**
Column	VF WAX (polar) 60 m * 0.25 mm * 0.25 µm	Injection	1.0µL
Vector gas	Helium (30 psis/FID)	Sample dilution	10% in hexane



Constituants	TR (min)	%	Constituants	TR (min)	%
1,8-Cineole	12.68	62.08	Copacamphene	35.69	0.18
α-Pinene	6.51	12.66	Menthatriene isomer	14.2	0.17
Aromadendrene	35.41	6.10	1,3,8-*p*-Menthatriene	12.73	0.16
Limonene	12.05	3.28	ß-Selinene	41.74	0.16
trans-Pinocarveol	37.87	1.96	γ-Terpinene	14.07	0.15
allo-Aromadendrene	37.49	1.49	α-*p*-Dimethylstyrene	24.86	0.15
α-Terpineol	40.73	1.48	Sesquiterpene	35.06	0.14
p-Cymene	15.25	1.19	Selinene isomer	34.86	0.13
α-Terpinyl acetate	40.52	0.97	ß-Caryophyllene	34.51	0.12
Globulol	61.28	0.65	Camphene	7.43	0.11
Pinocarvone	32.56	0.50	Terpinolene	15.91	0.11
trans-p-Mentha-1(7),8-dien-2-ol	46.17	0.39	α-Cubebene	26.86	0.11
α-Phellandrene	10.56	0.38	Sesquiterpene	40.28	0.11
Calarene	34.19	0.33	Viridiflorol	61.64	0.11
ß-Pinene	8.56	0.32	ß-Myrcene	10.43	0.10
epi-Globulol	58.12	0.25	Pinol	18.08	0.10
Terpinen-4-ol	35.02	0.23	ß-Pinocamphone + Sesquiterpene	31.25	0.10
α-Gurjunene	30.5	0.22	Gurjunene isomer	38.02	0.10
cis-p-Mentha -1(7),8(9)-dien-2-ol	51.25	0.21	Aromatic compound	42.06	0.10

A7. Lemon (*Citrus limon* (L.) Osbeck)

Instrument	GC/FID Agilent 6890	T° program (°C)	60°C (5') > 250°C (15') : 3°C/min
Column	VF WAX (polar) 60 m * 0.25 mm * 0.25 µm	Injection	1.0µL
Vector gas	Helium (30 psi/FID)	Sample dilution	10% in hexane



Compound	TR (min)	%
Limonene	13.63	65.39
ß-Pinene	9.97	13.61
γ-Terpinene	15.48	9.48
α-Pinene	7.4	2.28
Sabinene	10.36	2.08
ß-Myrcene	11.88	1.56
Geranial	36.93	1.46
Neral	34.87	1.02
p-Cymene	16.49	0.64
α-Thujene	7.48	0.46
Geranyl acetate	37.81	0.41
ß-Phellandrene	13.85	0.29
Neryl acetate	36.59	0.26
Terpinolene	17.06	0.25
α-Terpinene	12.59	0.15

A8. Clove (*Syzygium aromaticum* (L.) Merr. & L.M. Perry)

Compound	%
ß-Caryophyllene	3.61
Eugenol	83.38
Eugenyl acetate	10.48

A9. Mandarin (*Citrus reticulata* Blanco)

Compound	%
α-Pinene	2
ß-Pinene	1.4
Limonene	74.3
γ-Terpinene	17.3
Myrcene	1.8
α-Sinensal	0.16
Dimethyl anthranilate	0.35