Progress in Essential Oils

Brian M. Lawrence, Consultant

Niaouli Oil

Niaouli is the common name given to the oil obtained from *Melaleuca quinquinervia* (Cav.), S.T. Blake, a member of the Myrtaceae family and a sister species of the popular *Melaleuca alternifolia* (Maiden et Betche) Cheel or tea tree. *Melaleuca quinquinervia* is a small- to medium-sized tree that has white to grayish papery bark (characteristic of the Myrtaceae) and a turgid, leathery lanceolate-elliptic leaves. It can be found growing wild all along the east coast of Australia from Sydney northward. It can also be found in its wild habitat in southern Papua New Guinea and in New Caledonia. According to Rasoanaivo and De La Corce (1998), M. quinquinervia is native to the Moluccas Islands. It was introduced into Madagascar more than a century ago and it has now become established in the swampy areas along the eastern coastal region where it can be found in abundance (Rasonaivo and De La Corce, 1998).

Niaouli oil is produced from the leaves, twigs and small branches by steam and water distillation in which the steam is generated from water being boiled beneath the plant material packed on a perforated grid within the still above the water level.

The main center of production for niaouli oil has always been New Caledonia and the center of production there has been located around Gomen, which is why niaouli oil is also known in France as "gomenol." The annual production of niaouli oil varied from 15–20 tonnes prior to World War II. Since then, the oil production quantities have varied more widely from a high of ca. 30 tonnes to a low of <0.1 tonne. Current world production of niaouli oil is close to 1 tonne. Almost all of the current world production of niaouli oil is in Madagascar (Lawrence, 1999).

Turning now to the chemical composition of niaouli oil, in 1994 Ramanoelina et al. reported the results of the analysis of 144 samples of oil produced from leaves harvested at different times of the year from 48 trees randomly selected in eastern Madagascar in the vicinity of Toamasina. Of the more than 45 constituents identified by both GC/MS and retention index, the oils were grouped together into four chemotypes, as shown in **T-1**.

According to Ramanoelina et al. (1994) six chemotypes of *M. quinquinervia* have been reported to exist. They are as follows:

1. 1,8-cineole (37.0%)
2. viridiflorol (20.0%)
3. viridiflorol (48.0%)
4. (E)-nerolidol (87.0%)
5. (E)-nerolidol (74–95%) + linalool (14–30%)
6. 1,8-cineole (10–75%) + viridiflorol (13–66%) + α-terpineol (0.5–14%) + β-caryophyllene (0.5–28%)

The first four of these were found to occur in Madagascar while the last two were found in Australia and New Guinea (Ireland et al., 2002). This reviewer believes that the above-listed chemotypes are really not chemotypes but variations of oil composition within the same one or two chemotypes.

Brophy and Doran (1996) reported their results of analyses of oils produced in the laboratory from leaf material of *M. quinquinervia* (niaouli). The authors determined that the oils could be divided up into two chemotypes. Chemotype 1, which was found to be rich in (E)-nerolidol, possessed the following major components:

1,8-cineole (0.2%)
linalool (0.1%)
benzaldehyde (0.1%)
β-caryophyllene (0.1%)
β-farnesene[*] (0.1%)
(E)-nerolidol (95.0%)
(E,E)-farnesol (0.2%)

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[*] correct isomer not identified.

Chemotype 2, which was rich in 1,8-cineole, possessed the following composition:

α-pinene (2.0%)
β-pinene (0.8%)
sabinene (0.1%)
myrcene (1.2%)
α-phellandrene (0.1%)
α-terpinene (0.5%)
limonene (0.8%)
1,8-cineole (64.9%)
γ-terpinene (0.7%)
p-cymene (0.4%)
terpinolene (0.5%)
δ-elemene (0.1%)
benzaldehyde (0.1%)
α-gurjunene (0.1%)
terpinen-4-ol (0.2%)
β-caryophyllene (1.3%)
aromadendrene (1.7%)
α-bulnesene (0.1%)
allo-aromadendrene (0.3%)
δ-terpineol (9.9%)
viridiflorene (1.5%)
bicyclogermacrene (0.3%)
α-farnesene[*] (0.1%)
ledol (0.2%)
globulol (3.4%)
viridiflorol (0.2%)
spathulenol (0.3%)

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[*] correct isomer not identified

T-1 . Niaouli oil chemotypes

1. Oils rich in 1,8-cineole with varying amounts of α-terpineol
2. Oils rich in viridiflorol with varying amounts of 1,8-cineole and α-terpineol
3. Oils rich in (E)-nerolidol with varying amounts of linalool
4. Oils rich in 1,8-cineole with varying amounts of monoterpene hydrocarbons

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Trace amounts (<0.1%) of camphene, p-cymene, linalool, α-terpinyl acetate, δ-cadinene, γ-cadinene, a calamenene isomer, palustrol and a nerolidol isomer.

Brophy and Doran also noted that a variant of the 1,8-cineole-rich chemotype possessed the following major components:

α-pinene (10%)
benzaldehyde (0.2%)
limonene (2.5%)
β-caryophyllene (0.4%)
viridiflorene (1.0%)
viridiflorol (80.0%)
β-eudesmol (0.2%)
γ-eudesmol (0.2%)
α-eudesmol (0.2%)

A commercial sample of niaouli oil was found by Christoph et al. (2001) to contain the following constituents:

α-pinene (9.2%)
β-pinene (0.3%)
limonene (5.6%)
1,8-cineole (53.3%)
p-cymene (0.1%)
β-caryophyllene (0.1%)
α-humulene (0.2%)
α-terpineol (29.5%)

Trace amounts (<0.05%) of (E)-nerolidol and viridiflorol were also found in this oil.

Trilles et al. (2006) collected leaves of niaouli from seven different locations in New Caledonia. The oil contents ranged for the 133 samples collected from 0.5–2.5%. Analysis of these oils by GC-FID and GC/MS revealed that the 42 constituents characterized ranged as follows:

α-thujene (0–9.7%)
α-pinene (0–27.7%)
camphene (0–0.3%)
benzaldehyde (0–0.9%)
β-pinene (0.1–5.5%)
myrcene (t–2.0%)
α-phellandrene (0–1.9%)
δ-3-carene (0–0.4%)
α-terpinene (0–6.7%)
p-cymene (0–40.0%)
limonene (0.1–15.0%)
1,8-cineole (0.1–76.3%)
(E)-β-ocimene (0–0.3%)
γ-terpinene (0–32.4%)
terpinolene (t–19.2%)
terpinen-4-ol (0–5.0%)
α-terpineol (0–24.5%)
methyl thiobenzoate (0–0.3%)
α-terpinyl acetate (0–11.4%)
α-cubebene (0–0.3%)
α-gurjunene (0–t)
isocaryophyllene (0–2.1%)
β-caryophyllene (0–6.5%)
aromadendrene (0–0.4%)
α-humulene (0–0.2%)
allo-aromadendrene (0–1.2%)
ar-curcumene (0–0.6%)
β-selinene (0–1.4%)
ledene[a] (0–1.1%)
δ-cadinene (0–0.4%)
cis-calamenene (0–0.1%)
γ-cadinene (0–0.2%)
palustrol (0–t)
caryophyllene oxide + globulol (0–2.4%)
viridiflorol (0–67.4%)
guaiol (0–21.5%)
ledol[b] (0–8.8%)
10-epi-γ-eudesmol (0–22.6%)
γ-eudesmol (0–0.9%)
β-eudesmol (0–0.3%)
α-eudesmol (0–4.7%)
bulnesol (0–1.2%)
farnesol° (0–t)

° correct isomer not identifies
t = trace (0.05%)
[a] probably misidentification of viridiflorene
[b] probably misidentification of viridiflorol

Three hundred leaf samples were collected from 75 randomly chosen niaouli trees found along the road between Sémé and the Port-Novobridge (South Benin). Oils produced from 300–500 g of leaves were subjected to GC-FID and GC/MS analyses by Gbenou et al. (2007). The range of constituents found in these oils was as follows:

α-pinene (1.5–12.5%)
camphene (0.2–0.8%)
benzaldehyde (t–0.2%)
β-pinene (0.7–2.7%)
myrcene (t–0.8%)
α-terpinene (0.1–0.2%)
p-cymene (0.3–1.0%)
limonene (1.6–7.6%)
1,8-cineole (2.6–60.0%)
(E)-β-ocimene (t–0.2%)
γ-terpinene (t–1.0%)
terpinolene (0.1–0.3%)
α-phellandrene (t)
linalool (0.1–0.5%)
α-fenchyl alcohol (0.2–0.7%)
cis-sabinene hydrate (0.2–0.3%)
camphor (0.2–0.4%)
terpinen-4-ol (0.3–1.8%)
α-terpineol (1.1–15.0%)
α-terpinyl acetate (t–4.4%)
β-caryophyllene (t–1.7%)
α-himachalene (t–0.3%)
α-humulene (0.1–0.6%)
allo-aromadendrene (t–0.3%)
viridiflorene (0.2–1.1%)
γ-cadinene (t–1.0%)
δ-cadinene (t–0.1%)
ledol (0.8–1.5%)
(E)-nerolidol (t–3.2%)
spathulenol (t–3.6%)
caryophyllene oxide (0.4–0.9%)
viridiflorol (10.5–62.7%)
guaiol (t–0.2%)
γ-eudesmol (t–0.1%)
T-cadinol (0.1–0.4%)
β-eudesmol (0.1–0.4%)
α-eudesmol (0.1–0.3%)
α-cadinol (t–2.1%)
5-epi-paradisol (t–0.1%)
13-isopropylpodocarpa-8,13-dien-15-ol (t–0.2%)

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t = trace (<0.05%)

Further, Gbenou et al. used principal component analysis (PCwA) to examine the data. The conclusion drawn from this study shows that the authors have determined that the Benin niaouli oil exists in three chemotypic forms as shown in **T-2**. However, the oils are more likely of the same chemotype with varying levels of 1,8-cineole and viridiflorol.

An oil of *M. quinquinervia* produced in the laboratory from leaves collected in an arboretum in Viçosa (Minas Gerais, Brazil) was analyzed by Silva et al. (2007). The main components of this oil were found to be:

α-pinene (4.7%)
1,8-cineole (7.2%)
α-terpineol (2.2%)
caryophyllene oxide (3.5%)
viridiflorol (71.0%)

Ramanoelina et al. (2008) used GC-FID and GC/MS to examine the composition of 159 samples of niaouli oil produced in Madagascar. The range in composition of these oils can be seen as follows:

α-pinene (0.1–16.9%)
β-pinene (t–4.5%)
myrcene (t–2.5%)
limonene (0.1–11.7%)
1,8-cineole (t–71.1%)
γ-terpinene (t–7.4%)
p-cymene (t–5.0%)
terpinolene (t–3.7%)
benzaldehyde (t–2.5%)
linalool (0.1–6.6%)
α-gurjunene (t–3.1%)
β-caryophyllene (0.2–5.9%)
terpin en-4-ol (t–4.6%)
viridiflorene (t–2.6%)
α-terpineol (t–12.3%)
δ-cadinene + γ-cadinene (t–1.6%)
caryophyllene oxide (t–1.3%)

ledol (0.1–7.8%)
(E)-nerolidol (0.1–95.0%)
viridiflorol (0.1–36.3%)
α-muurolol (t–1.6%)

t = trace (<0.05%)

T-2. Average percentage composition of the three niaouli oil chemotypes found in Benin

Compound	Type 1	Type 2	Type 3
α-pinene	5.3	5.5	5.9
β-pinene	1.4	1.4	1.6
p-cymene	0.5	0.5	0.5
limonene	5.6	5.4	4.4
1,8-cineole	49.6	33.2	12.4
γ-terpinene	0.6	0.7	0.8
linalool	0.2	0.3	0.3
terpinen-4-ol	0.9	0.9	1.2
α-terpineol	10.7	7.8	4.1
α-terpinyl acetate	0.6	0.9	0.8
β-caryophyllene	0.2	0.8	1.0
α-humulene	0.1	0.2	0.2
viridiflorene	0.6	0.9	0.7
ledol	1.1	1.5	1.3
(E)-nerolidol	0.5	0.5	0.5
spathulenol	0.3	0.5	4.2
caryophyllene oxide	0.7	1.3	1.8
viridiflorol	16.5	32.0	50.3
T-cadinol	0.4	0.8	1.1
α-cadinol	0.3	0.5	0.7

T-3. Percentage range of the constituents in the three chemotypes of Madagascan niaouli oil

Compound	Type 1	Type 2	Type 3
α-pinene	2.8–16.9	1.3–10.3	0.1–8.4
β-pinene	1.0–4.5	1.9–3.5	t–2.4
myrcene	0.1–2.1	0.2–2.5	t–0.4
limonene	4.8–11.7	2.6–6.3	0.1–7.7
1,8-cineole	34.9–71.1	8.6–31.0	t–16.2
γ-terpinene	0.3–7.4	0.4–6.6	t–1.4
p-cymene	0.1–5.0	0.5–2.5	t–0.5
terpinolene	0.1–3.7	0.2–3.6	t–0.3
benzaldehyde	t–0.6	0.1–2.5	0.1–1.0
linalool	0.1–1.0	0.1–6.6	0.1–1.0
α-gurjunene	t–0.3	t–3.1	t–0.7
β-caryophyllene	0.2–3.4	2.1–5.0	1.2–5.9
terpinen-4-ol	0.1–2.0	0.5–5.0	t–1.4
viridiflorene	0.1–2.0	0.8–2.6	t–0.7
α-terpineol	3.2–12.3	4.9–11.3	0.1–3.8
δ-cadinene + γ-cadinene	t–0.8	0.2–1.6	0.1–0.2
caryophyllene oxide	t–1.1	0.2–1.0	0.2–1.3
ledol	0.1–1.7	1.1–7.8	0.2–1.6
(E)-nerolidol	0.1–20.2	4.7–24.2	43.8–95.0
viridiflorol	1.6–16.1	17.1–36.3	0.1–11.8
α-muurolol	t–1.1	0.1–1.6	0.1–0.5

t = trace (<0.05%)

Ramanoelina et al. also examined their data using PCA and determined the existence of three chemotypes within the 159 samples of Madagascan niaouli oil. A summary of the component range for the three chemotypes (1,8-cineole-rich, viridiflorol-rich and (E)-nerolidol-rich) can be seen in **T-3**.

Nine samples of niaouli oil produced from areas in both north and south New Caledonia were examined for their major constituents by Radioas et al. (2008). The oils that were produced in yields of 0.75–2.75% were found to possess the following range of major components:

1,8-cineole (7.5–41.1%)
α-terpineol (0.1–11.5%)
viridiflorol (12.3–47.7%)

A commercial sample of New Caledonia niaouli oil was found to contain:

1,8-cineole (44.5%)
α-terpineol (4.2%)
viridiflorol (3.8%)

The authors noted that this commercial oil was incomplete because it was almost devoid of sesquiterpenoid compounds.

Lee et al. (2008) screened a number of oils produced from plants of the Myrtaceae family. Among these oils was a sample of niaouli oil purchased in Australia. Using GC-FID and GC/MS, the oil was determined to contain:

allyl isothiocyanate[a] (0.1%)
α-pinene (3.1%)
β-pinene (1.1%)
myrcene (0.3%)
p-cymene (0.6%)
1,8-cineole (35.0%)
limonene (1.1%)
γ-terpinene (0.4%)
terpinolene (0.1%)
linalool (15.2%)
terpinen-4-ol (1.3%)
α-terpineol (7.2%)
β-caryophyllene (0.7%)
α-humulene (0.2%)
(E)-nerolidol (24.2%)

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[a] probably a contaminant as it is not a niaouli oil component

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Cornmint Oil

Simizu (1991 reported that over the years the cornmint grown for oil production and ultimate menthol isolation in Japan has changed. The cultivars grown in Japan were Akamaru (1924), Hokoshin (1938), Manyo (1953), Suzukaze (1954), Ouba (1962), Honyo (1965) and Ayanarin (1968). He also reported that Higashima and Sakata (1981) determined two main constituents of dementholized cornmint oil of Japanese and Chinese origins. These results are found in **T-4**. Furthermore, he reported the results of an analytical evaluation of the oils produced from six experimental clones of *Mentha canadensis* grown in Japan (**T-5**).

T-4. Percentage composition of dementholized cornmint oil of Japanese and Chinese origins

Compound	Japanese oil	Chinese oil
limonene	1.2	3.2
3-octanol	2.4	1.9
menthone	15.1	23.5
isomenthone	7.9	14.7
neomenthol	3.7	7.7
menthol	25.7	35.2
menthyl acetate	16.7	4.4
pulegone	2.1	1.4
piperitone	12.1	2.6

T-5. Percentage composition of some Japanese oils of *Mentha canadensis* (cornmint)

Compound	1	2	3	4	5	6
α-pinene	0.3	0.8	0.5	0.4	0.7	0.7
β-pinene	1.2	2.2	0.9	1.9	1.6	2.3
limonene	1.7	1.8	1.1	1.6	0.5	0.9
1,8-cineole	0.1	0.1	0.1	0.5	0.3	0.5
3-octanol	0.2	0.7	0.4	0.3	0.3	0.2
menthofuran	-	-	-	0.7	0.5	0.3
menthone	9.8	9.6	3.7	12.2	16.8	15.5
isomenthone	1.4	1.1	0.4	0.9	2.8	2.4
menthyl acetate	-	-	-	2.1	1.4	1.9
neomenthol	2.6	2.6	3.1	2.0	2.2	1.3
menthol	81.0	79.1	86.0	72.9	69.4	71.3
pulegone	1.1	0.7	2.0	1.6	1.5	1.1
piperitone	0.4	0.5	0.9	0.3	0.5	0.4

1–6 are oils produced from experimental clones of *M. canadensis* produced in Japan.

Singh et al. (1998) studied the vatriability found in 38 genotypes of cornmint in the germplasm collection at the Central Institute of Medicinal and Aromatic Plants (Lucknow, U.P., India). They examined the herbage yield per hectare, the oil yield in kilograms per hectare, the oil yield per plant as well as the menthol, menthone, isomenthone, neomenthol and menthyl acetate contents of the oils. Using extrapolated data, the following ranges were found:

Herbage yield per hectare: 8.4–18.3 mt
Oil yield per hectare: 37.6–136.0 mt
Oil content per plant: 0.36–0.97%
Menthol: 70.5–84.3%
Menthone: 1.6–11.0%
Isomenthone: 1.1–7.6%
Neomenthol: 0.3–8.8%
Menthyl acetate: 0.6–7.3%

Using principal component analysis, the authors found that the 38 genotypes could be grouped into eight clusters. The problem with extrapolating data obtained from a limited number of plants (as was done by Singh et al.), is that it is rarely an accurate picture of the real data that can be obtained from a commercial planting of a genotype. For example, if one

T-6. Comparative percentage of selected components of the oils of Indian cornmint cultivars

	Cornmint cultivars				
Compound	**Shivalik**	**Kalka**	**Gomti**	**Himalaya**	**Kosi**
menthone	7.7	4.5	13.7	6.3	4.9
isomenthone	4.2	2.1	3.2	2.5	2.6
menthyl acetate	3.7	4.4	5.7	7.6	9.1
neomenthol	2.0	2.2	2.0	2.1	2.3
menthol	73.7	75.3	68.1	71.4	71.0

T-7. Comparative percentage composition of the oils of the 'Shivalik' cultivar of cornmint grown in two different Indian regions

Compound	**Hyderabad (AP)**	**Sambhal (UP)**
α-pinene	0.6	0.6
sabinene + 1-octen-3-ol	0.2	0.3
β-pinene	0.5	0.7
myrcene + 3-octanol	1.1	1.1
limonene + 1,8-cineole	2.0	1.0
linalool	0.3	0.1
menthone	10.2	7.6
isopulegol	-	0.1
isomenthone	2.0	4.9
neomenthol	1.6	2.1
menthol	70.5	75.5
isomenthol + α-terpineol	0.1	1.2
pulegone	6.6	0.4
piperitone	0.5	0.3
menthyl acetate	-	1.7
β-caryophyllene	0.4	0.5
germacrene D	0.5	0.6

AP = Andhra Pradesh
UP = Uttar Pradesh

examines the major components of the commercially grown cornmint cultivars shown in **T-6** (Bahl et al., 2000), it can be seen that the component level listed by Singh et al. for the oil of the 'Shivalik' cultivar were:

menthone (8.2%)
isomenthone (5.8%)
menthyl acetate (2.1%)
neomenthol (3.1%)
menthol (76.5%)

These were slightly dissimilar to the data reported by Singh et al. (1998). The high oil-yielding genotypes that were rich in menthol reported by Singh et al. were unfortunately not identified by any cultivar name other than 'Shivalik.'

Rajeswara Rao et al. (1999) compared the composition of cornmint oils produced from the 'Shivalik' cultivar that were grown in different regions of India. The results of this study are shown in **T-7**.

Ranade (2005a) reported that the main constituents of a crude cornmint oil were as follows:

β-pinene (1.3%)
1,8-cineole (0.8%)
(Z)-3-hexenol (0.2%)
limonene (4.1%)
menthone (13.8%)
menthol (65.0%)
linalool (0.8%)
menthofuran (0.1%)
menthyl acetate (4.7%)
neomenthol (2.2%)
neoisomenthol (1.2%)
3-octanol (0.4%)
pulegone (1.1%)
piperitone (1.8%)
piperitone oxide[*] (0.3%)

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[*] correct isomer not identified

It is quite surprising that Ranade did not identify any isomenthone in this oil as the isomenthone/menthone ratios are used to differentiate Indian and Chinese cornmint oils.

The composition of a dementholized oil of cornmint of Indian origin was reported by Ranade (2005b) to possess the following composition:

α-pinene (0.7%)
β-pinene (1.0%)
sabinene (0.4%)
myrcene (0.1%)
α-terpinene (0.3%)
limonene (0.7%)
1,8-cineole (7.4%)
(Z)-β-ocimene (0.3%)
(E)-β-ocimene + γ-terpinene (0.6%)
terpinolene (0.2%)
3-octanol (0.2%)
trans-sabinene hydrate (1.8%)
menthone (16.0%)
menthofuran (0.1%)
isomenthone (2.5%)
menthyl acetate (2.0%)
neomenthol (4.1%)
β-caryophyllene (2.2%)
menthol (46.2%)
pulegone (0.1%)
piperitone (0.8%)

The volatile concentrate of dried cornmint leaves that were purchased from an oriental herbal market in Seoul (Korea) was analyzed by Ka et al. (2005). The volatiles characterized in this concentrate were:

neomenthol (717)[a]
menthol (18,379)
2-phenethyl alcohol (138)
germacrene D-4-ol (53)
eugenol (145)
α-cadinol (71)
thymol (126)
α-muurolol (208)
menthyl acetate (2,439)
methyl jasmonate[†] (38)
menthone (2,323)
cis-piperitone oxide (282)
piperitone (4,656)
2-hydroxypiperitone (40)
(Z)-jasmone (65)
germacrene D (61)
calamenene[*] (40)
dihydroactinodiolide (66)
(Z)-asarone[b] (32)

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[a] mg/g dried leaves
[†] doubtful constituent
[b] probably contaminant from *Asarum gramineus* leaves

A sample of cornmint oil produced in Nepal was screened for its antimicrobial properties by Yozon et al. (2005). The constituents identified in this oil were:

α-pinene (1.5%)
β-pinene (1.5%)
limonene (1.9%)
menthone (13.9%)
isomenthone (9.5%)
isopulegol (1.5%)
neomenthol (4.0%)
β-caryophyllene (1.1%)
neoisopulegol† (1.4%)
menthol (53.0%)
germacrene D (1.8%)
geraniol (1.5%)

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† probably misidentification of neoiso(iso)pulegol

Mestri (2006) compared the composition of cornmint oil of Brazilian (none is currently produced) Chinese and Indian origins. The comparative data reported can be seen in **T-8**. It was also reported that the constituents previously identified in cornmint oil (no references given) could be grouped as follows:

hydrocarbons (21)
alcohols (24)
esters (21)
aldehydes (6)
ketones (12)
acids (7)
phenols (1)
nitrogen heterocyclic compounds (3)
sulfur compounds (1)
ethers/epoxides (9)

T-8. Comparative main components of cornmint oil of different origins

Compound	Brazilian	Chinese	Indian
monoterpene hydrocarbons	6.8	5.5	6.0
menthone	14.8	11.1	6.1
isomenthone	4.1	4.0	2.7
menthyl acetate	2.4	0.8	2.0
neomenthol	2.6	2.6	2.8
menthol	65.1	72.1	75.5
isomenthol	0.4	0.8	0.8
piperitone	1.9	0.9	1.1
pulegone	1.2	0.9	0.5

Gassonmeier (2006) identified L-menthyl lactate in dementholized cornmint oil of Indian origin. He postulated that lactic acid producing microorganisms formed lactic acid when fresh cornmint herbage is stored under humid conditions for a prolonged time period thereby allowing some fermentation. The author confirmed that when lactic acid and dried peppermint leaves were mixed and subjected to hydrodistillation using a Clevenger-type system, menthyl lactate could be detected in the oil.

Lawrence (2007) reported that the constituents (196) that had been previously identified in cornmint oil prior to 2006 were as follows:

hydrocarbons (41)
alcohols (58)
esters (30)
aldehydes (13)
ketones (18)
acids (13)
phenols (4)
nitrogen heterocyclic compounds (5)
sulfur compounds (2)
ethers and epoxides (9)
lactones (3)

Liang et al. (2007) compared the composition of an oil and a supercritical fluid CO_2 extract of cornmint (known in China as *Mentha haplocalyx* Briq., which is a synonym of *M. canadensis*). The results of this study are shown in **T-9**.

T-9. Comparative percentage composition of an oil and supercritical fluid CO_2 extract of cornmint grown in China

Compound	Oil	Extract
α-pinene	t	0.4
camphene	t	0.2
β-pinene	0.1	0.5
myrcene	0.1	0.3
3-octanol	1.4	1.6
limonene	0.2	0.7
isopulegol	-	0.2
menthone	12.7	13.5
isomenthone	3.7	4.3
menthol	69.4	61.8
α-terpineol	0.6	-
(Z)-3-hexenyl valerate	0.2	0.3
pulegone	0.9	0.8
piperitone	3.4	2.2
β-bourbonene	0.3	0.6
β-caryophyllene	1.1	1.5
germacrene D	1.7	3.1
bicyclogermacrene	0.3	-
γ-elemene	-	0.5
δ-cadinene	0.2	-
spathulenol	0.2	-
hexadecanoic acid	0.1	0.3
phytol	0.1	0.3
linoleic acid	-	0.3
hentriacontane	-	0.7

Pant et al. (2008) and Malik et al. (2008) used GC/MS as their only method of analysis of a sample of cornmint oil produced by hydrodistillation of plants harvested in Selaqui (Uttarakhand, India). The components characterized in this oil were:

2,5-diethyltetrahydrofuran* (<0.1%)
α-pinene (0.1%)
camphene (0.1%)
β-pinene (0.7%)
3-octanol (0.4%)
myrcene (0.4%)
m-cymene† (0.1%)
limonene (2.3%)

T-10. Comparative percentage composition of crude and dementholized Indian cornmint oil

Compound	Crude oil	Dementholized oil
α-pinene	0.4	2.0
β-pinene	0.5	2.3
sabinene	0.2	-
myrcene	0.2	1.3
p-cymene	-	0.2
limonene	t	4.5
1,8-cineole	0.2	0.8
(Z)-3-hexenol	0.6	0.1
3-octanol	-	1.6
menthone	4.1	23.9
isomenthone	2.7	13.8
linalool	-	0.1
neomenthol	1.1	4.7
menthol	78.9	28.1
β-caryophyllene	0.5	0.5
caryophyllene oxide	-	0.1

t = trace (<0.1%)

1,8-cineole (0.5%)
p-menthan-3-one[‡] (6.9%)
p-menthone [‡‡] (5.9%)
menthol (65.2%)
menthol[a] (0.4%)
(Z)-3-hexenyl isovalerate (0.3%)
carvone (0.4%)
piperitone (0.3%)
geranial (0.2%)
menthol[†]
menthyl acetate[†]

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[a] menthol can only occur once; it is possible that this is a menthol isomer

[*] correct isomer not identified

[†] incorrect identification based on GC elution order

[‡] this should be menthone

[‡‡] this should be isomenthone

The authors also erroneously characterized trace amounts of 1-phenylhexene, a phellandrene, δ-3-carene and carvone because they used computer matching of mass spectral patterns and did not use any other techniques to confirm their results.

A commercial sample of a dementholized oil of cornmint was the subject of analysis by Jirovetz et al. (2009). Using a combination of GC techniques the authors determined that the oil had the following composition:

α-thujene (0.1%)
α-pinene (0.1%)
camphene (0.1%)
heptanol (0.1%)
sabinene (0.4%)
β-pinene (1.3%)
myrcene (0.5%)
3-octanol (1.1%)
p-cymene (0.1%)
limonene (4.3%)
1,8-cineole (2.7%)
(E)-β-ocimene (0.1%)
γ-terpinene (0.1%)
octanol (0.1%)
terpinolene (0.2%)
linalool (0.2%)
3-nonanol (0.1%)
isopulegol (1.2%)
menthone (20.4%)
isomenthone (9.1%)
neomenthol (4.0%)
menthol (41.2%)
α-terpineol (0.6%)
neoisomenthol (0.6%)
pulegone (1.6%)
carvone (0.3%)
(Z)-3-hexenyl isovalerate (0.4%)
piperitone (1.2%)
(E)-anethole (0.2%)
isomenthyl acetate (0.1%)
menthyl acetate (4.6%)
β-bourbonene (0.3%)
β-elemene (0.1%)
β-caryophyllene (0.9%)
germacrene D (0.2%)
δ-cadinene (0.2%)

It should be noted that prior to 2006 (Lawrence, 2007) 3-nonanol and (E)-anethole have not been found as constituents of cornmint oil. In addition, Jirovetz et al. characterized trace amounts (<0.1%) of (Z)-3-hexenol, hexanol, a pulegol isomer, an isopulegone isomer and nonyl acetate in the oil.

Oils produced from three leaves and inflorescences of a Russian cultivar of *Mentha canadensis* were by Kurilov et al. (2009). The components characterized in these oils, which were found to be very similar, were as follows:

α-pinene (0.6%)
β-pinene (0.6%)
p-cymene (0.1%)
limonene (1.1–2.2%)
menthone (25.9–31.2%)
menthol (30.7–33.2%)
pulegone (0.5–1.9%)
piperitone (1.6–1.7%)
menthyl acetate (24.9–29.5%)
β-bourbonene (0.5–0.6%)
β-cubebene (0.5%)
aromadendrene (2.3–2.5%)
γ-muurolene (0.6–0.7%)
germacrene D (0.3%)
α-muurolene (1.4–1.6%)
δ-cadinene (0.3%)
spathulenol (0.1–0.2%)
T-cadinol (0.2%)

Zheljazkov et al. (2010) examined the potential of growing cornmint in Verona, Mississippi. They found that the oil content of the two cultivars grown ranged from 0.15–0.38% on a fresh weight basis and the menthol content ranged from 67–78%. Although the authors showed that cornmint could be successfully grown in Mississippi, an economic evaluation on the cost effectiveness of growing and producing oil from cornmint was not conducted. Also, it would appear that with the low oil yields reported the potential yield per acre could not compete with the typical yields per unit area in India.

Ahmad et al. (2011) reported that the main components found in crude Indian cornmint oil and dementholized cornmint oil were as shown in **T-10**.

A sample of dementholized cornmint oil of Indian origin that was screened for its insect activity against the cotton leaf worm by Pavela (2012) was determined by GC FID and GC/MS to possess the following composition:

β-pinene (1.1%)
limonene (2.5%)
isopulegol (1.2%)
menthone (25.7%)
isomenthone (12.1%)
menthol (45.7%)
neomenthol (1.3%)
pulegone (2.5%)
piperitone (1.8%)
menthyl acetate (4.3%)

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T-11. Comparative percentage composition of *Cormbia citriodora* oil and the oil extracted from the distillation water

Compound	Steam-distilled	Distillation water
α-thujene	0.2	t
sabinene	0.4	0.2
myrcene	0.2	-
α-phellandrene	t	-
α-terpinene	0.1	-
p-cymene	0.5	-
limonene	0.2	0.1
1,8-cineole	0.2	-
(Z)-β-ocimene	t	0.1
(E)-β-ocimene	0.2	0.1
cis-linalool oxide[f]	-	0.1
trans-linalool oxide[f]	0.1	0.1
linalool	0.6	1.0
citronellal	70.3	-
isopulegol	6.7	53.0
borneol	0.8	10.0
menthol	0.3	5.3
terpinen-4-ol	0.1	0.6
α-terpineol	-	0.8
citronellol	8.8	0.1
neral	0.1	6.9
geraniol	0.1	1.4
geranial	t	0.1
unknown	1.0	10.3
citronellyl acetate	1.3	t
eugenol	0.1	4.6
geranyl acetate	0.1	0.2
β-elemene	-	0.9
β-caryophyllene	2.6	0.1
aromadendrene	0.1	-
α-humulene	0.1	-
γ-cadinene	t	-
δ-cadinene	0.1	-
caryophyllene oxide	t	-

t = trace (<0.05%)
[f] furanoid form

Eucalyptus citriodora Oil

Sood et al. (1987) determined the composition of an oil produced from the young leaves of *Cormbia citriodora* Hook. (syn. *Eucalyptus citriodora* Hook.) and found that it contained citronellal (32.0%) and citronellal (51.7%) as compared to 66.6% and 23.1%, respectively, found in an oil produced from old leaves.

Rajeswara Rao et al. (2003) used GC-FID, retention indices and peak enrichment to examine the composition of a steam-distilled oil of *C. citriodora* and compared it to an oil extracted from the distillation water using hexane. The

T-12. Percentage composition of *Eucalyptus citriodora* oils produced over a single season in Egypt

Compound	January oil	April oil	July oil	October oil
α-thujene	-	-	-	0.3
α-pinene	-	t	t	1.2
sabinene	0.3	-	-	-
myrcene	-	0.1	-	-
δ-3-carene	0.7	t	-	-
α-menthene*	-	-	-	4.1
1,8-cineole	3.5	-	0.4	t
β-phellandrene	0.4	-	-	-
linalool oxide*	2.4	t	-	-
terpinolene	-	-	t	0.2
citronellal	31.1	65.4	60.3	77.9
isopulegol	1.8	-	-	-
dihydrocitronellol†	-	-	1.5	-
citronellol	17.0	14.7	15.8	12.5
geraniol	-	-	t	-
isopulegyl acetate	-	t	-	-
hydroxycitronellol†	-	-	1.4	0.6
p-menth-1-en-9-ol	-	t	-	-
citronellyl acetate	3.8	14.2	9.8	2.1
geranyl acetate	-	-	2.1	-
β-caryophyllene	3.9	0.9	t	0.1
spathulenol	1.1	t	-	-
caryophyllene oxide	5.5	6.9	2.3	t
isospathulenol	-	-	0.1	-
guaiol‡	-	0.1	-	-
(E,E)-farnesol	-	0.1	-	-
farnesol*	-	t	0.2	-

t = trace (<0.1%)
* correct isomer not identified
† incorrect identification, compound not naturally occurring
‡ incorrect identification based on GC elution order

comparative composition of the two oils can be seen in **T-11**. Unfortunately, the authors did not report any yield data of the distilled oil or the distillate oil.

El-Zalabani et al. (2007) examined the seasonal variation in oil composition of *E. citriodora* leaves harvested from trees grown in Egypt. A summary of the findings of this study can be seen in **T-12**.

A sample of a Chinese commercial oil of *C. citriodora* was analyzed using GC-FID and GC/MS by Jirovetz et al. (2007). The constituents characterized in this oil were as follows:

isobutyl isobutyrate (0.1%)
α-pinene (0.9%)
camphene (0.1%)
sabinene (0.1%)
β-pinene (0.7%)
myrcene (0.1%)
α-phellandrene (0.1%)
p-cymene (0.1%)
limonene (0.2%)
1,8-cineole (0.5%)
β-phellandrene (0.2%)
citronellal (76.3%)
γ-terpinene (0.1%)
terpinolene (0.1%)
linalool (0.5%)
neoisopulegol (5.5%)
iso(iso)pulegol (2.8%)
neoiso(iso)pulegol (0.3%)
terpinen-4-ol (0.2%)
α-terpineol (0.1%)
citronellol (5.7%)
geraniol (0.4%)
citronellyl acetate (0.9%)
geranyl acetate (0.1%)
β-caryophyllene (1.2%)
longifolene† (0.3%)

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† incorrect identification

Vaknin et al. (2009) performed some pot-size studies on the effect of plant height, stem diameter, dry leaf biomass, essential oil content and oil composition of *C. citriodora*. Although there were effects on all the physical attributes, the oil composition was found to be fairly similar as can be seen as follows:

α-pinene (0.2–0.7%)
sabinene (0.1–0.2%)
β-pinene (1.0–1.6%)
myrcene (0.3–0.5%)
p-cymene (t–0.1%)
limonene (t–0.1%)
(E)-β-ocimene (t)
melonal[a] (0.1–0.2%)
γ-terpinene (t–0.2%)
terpinolene (t–0.1%)
linalool (0.2–0.4%)
neoisopulegol (t–1.9%)
citronellal (66.9–71.5%)
iso(iso)pulegol (1.3–2.3%)
neoiso(iso)pulegol (0.2–0.4%)
citronellol (11.6–15.9%)
geraniol (t–0.2%)
geranial (0.1–0.2%)
p-vinylguaiacol[a] (0.8–1.7%)
bicycloelemene (0.1–0.2%)
β-elemene (t)
citronellyl acetate (1.2–2.4%)
eugenol (0.6–1.0%)
neryl acetate (t)
geranyl acetate (0.2–1.1%)
2-phenethyl isobutyrate (0.1–0.2%)
β-caryophyllene (5.5–6.5%)
citronellyl propionate (t–0.1%)
(E)-isoeugenol (t–0.2%)
α-humulene (0.4%)
allo-aromadendrene (t)
bicyclogermacrene (0.2–0.3%)
geranyl isobutyrate (t)
spathulenol (t–0.1%)
caryophyllene oxide (0.2%)

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t = trace (<0.05%)
[a] also known as 2,6-dimethyl-5-heptenal

[b] also known as 2-methoxy-4-vinylphenol (which is a major contribution to the odor of buckwheat)

A steam-distilled oil that was produced by hydrodistillation from *C. citriodora* leaves collected from N'Debougou (Mali) was analyzed by Traoré et al. (2009) using GC-FID and GC/MS. The oil composition was determined to be as follows:

α-pinene (1.3%)
myrcene (0.2%)
α-terpinene (0.1%)
p-cymene (0.3%)
limonene (0.1%)
citronellal (76.3%)
γ-terpinene (0.2%)
trans-rose oxide (0.3%)
terpinolene (0.5%)
cis-rose oxide (0.9%)

citronellol (7.3%)
citronellyl acetate (0.3%)
methyl eugenol (8.2%)
β-caryophyllene (0.4%)
aromadendrene (0.1%)
allo-aromadendrene (0.1%)
γ-gurjunene (0.3%)

A lab-distilled oil of *C. citriodora* was screened against the eggs, larvae and adult phases of *Lutzomyia longipalpis* (a Brazilian sandfly) which is the main vector for the chronic disease caused by protozoa of visceral leishmaniasis. The main components in the screened oil were determined by Maciel et al. (2010) to be:

α-pinene (1.1%)
limonene (0.8%)
isopulegol[a] (7.3%)
citronellal (71.8%)
isopulegol[b] (4.3%)
citronellol (2.9%)

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[a] neoisopulegol
[b] iso(iso)pulegol

Manguro et al. (2010) collected fresh leaves of *C. citriodora* (syn. *E. citriodora*) from Ethiopia and Kenya. Oils produced from these leaves were analyzed by GC-FID and GC/MS and their compositions can be found in **T-13**. Elaissi et al. (2011) examined the composition of seven oils produced from various *Eucalyptus* species grown in Tunisia. Using a combination of GC-FID and GC/MS, an oil of *E. citriodora* that was produced in 3.3% yield was found to possess the following composition:

α-pinene (23.6%)
α-fenchene (0.1%)
camphene (0.5%)
β-pinene (0.1%)
isoamyl acetate (0.1%)
α-phellandrene (0.1%)
limonene (2.5%)
β-phellandrene (0.6%)
1,8-cineole (54.1%)
γ-terpinene (1.2%)
p-cymene (2.8%)
terpinolene (0.5%)
isoamyl isovalerate (0.4%)
hexyl isobutyrate (0.1%)
p-cymenene (0.1%)
α-copaene (0.1%)
pinocarvone (1.6%)
p-cymen-8-ol (0.1%)
cis-p-mentha-1(7),8-dien-2-ol (0.2%)
ledol (0.7%)
globulol (0.1%)
spathulenol (0.1%)
carvacrol (0.1%)
α-eudesmol (0.1%)
β-eudesmol (0.1%)

T-13. Percentage composition of oils of *Eucalyptus citriodora* of Ethiopian and Kenyan origin

Compound	Ethiopian oil	Kenyan oil
(E)-β-ocimene	-	0.1
α-thujene	-	0.3
α-pinene	0.1	-
β-pinene	0.2	t
myrcene	t	0.1
1,8-cineole	t	0.3
limonene	-	0.1
γ-terpinene	-	0.1
terpinolene	0.1	0.1
linalool	0.2	0.3
isopulegol	4.7	3.7
citronellol	7.8	-
citronellal	76.4	84.9
carvacrol	-	4.5
α-terpinyl acetate	-	0.1
cuminaldehyde	-	4.2
β-caryophyllene	0.2	0.1
citronellyl acetate	1.9	2.5
geranyl acetate	t	-

t = trace (<0.1%)

Unfortunately, the authors misidentified the *Eucalyptus* species from which the oil was obtained because a 1,8-cineole-rich *E. citriodora* oil has never been found.

Rajeswara Rao et al. (2011) showed that when an oil of *E. citriodora* is exposed to water during storage, the minor changes in composition of the major constituents can be seen as follows:

citronellal	76.3% to 73.9%
isopulegol	4.9% to 6.8%
citronellol	5.5% to 5.1%
linalool	1.1% to 1.0%

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