Progress in Essential Oils

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Black and White Pepper Oil

Tewtrakul et al. (2000) produced an oil from black pepper (*Piper nigrum* L.) of Malaysian origin using hydrodistillation. This oil, which was produced from a generic black pepper, was analyzed by GC/MS, and its composition was determined to be as follows:

α-pinene (0.3%)
sabinene (2.4%)
myrcene (9.1%)
δ-3-carene (10.9%)
limonene (8.7%)
δ-elemene (2.5%)
α-copaene (3.8%)
β-elemene (1.6%)
β-caryophyllene (39.7%)
α-humulene (2.8%)
β-selinene (1.1%)
α-selinene (1.1%)
δ-cadinene (3.9%)
caryophyllene oxide (4.1%)

Nirmala Menon (2000) compared the chemical composition of oils produced from fresh and dried peppercorns. A summary of the results is presented in **T-1**. It should be pointed out that FP-1 and DP-1 were actually aroma concentrates that were produced by mashing either the fresh green peppercorns or dried peppercorns with water until becoming pastelike. After washing with water to remove soluble sugars, amino acids and other water-soluble materials, this fluid pastelike mix was passed through an Amberlite XAD-2 column. The neutral organic materials were removed from the XAD-2 column by eluting with pentane diethyl ether (1:1) mixture. After concentration to remove the solvents, the final aroma concentrates were subjected to GC/MS. As can be seen from the results, the oil produced from fresh green pepper was different from an oil produced from dried pepper mash. Similarly, the oils produced from fresh and dried pepper were also found to be different.

Oils produced by hydrodistillation from black pepper purchased at a local market in Mysore (India) and ground to various particle sizes, were obtained in yields of 1.48–2.92% (Mohan Rao, 2000). The composition of oils produced from ground pepper of particle diameters ranging 0.61–2.08 mm at temperatures of 30–31°C can be seen in **T-2.**

In India and other parts of the tropical world, there are numerous cultivars of black pepper grown (Lawrence, 1981) depending on the extrinsic factors experienced by the plant. These cultivars are mostly selections that have been found to grow well in a particular region. Most of the cultivars grown can be found in India. However, because of an intensive breeding program certain cultivars such as Paniyur types have been produced by other than selective means. Between 2000 and 2005 Nirmala Menon and co-workers have reported on the composition of a number of the most common cultivars [Nirmala Menon et al. (2000); Nirmala Menon (2002); Nirmala Menon et al. (2003), Nirmala Menon and Padmakumari (2005 a) and Nirmala Menon and Padmakumari (2005 b)]. A summary of the results of these analyses can be seen in **T-3.** The range of data relates to oils produced from black pepper cultivars over three consecutive seasons for most or only two consecutive seasons for the "Kuthiravally," "Uthirankotta" and "Kuching" cultivars. The "Kuching" cultivar originated from Sarawak, but was grown under the same conditions as all of the other cultivars grown in Kerala (India).

As can be seen, the major mono- and sesquiterpene hydrocarbons varied from one season to another. α-Pinene ranged from 1.7–14.6%, sabinene from 0–27.5%, β-pinene from 0–23.9%, δ-3-carene from 0–23.4%, limonene from 8.3–23.8% and β-caryophyllene from 6.4–52.9%.

Mathew (2001) reviewed pepper oil composition. Unfortunately, this author failed to review all of the published data available at the time. As a result, his review is scant at best.

An oil produced from black pepper grown in Holguin (Cuba) was analyzed using GC/MS by Pino et al. (2001). The following components were characterized in the oil:

α-pinene (3.1%)
camphene (0.1%)
m-cymene[†] (0.4%)
sabinene (0.1%)
myrcene (0.1%)
α-terpinene (7.3%)
p-cymene (0.8%)
1,8-cineole (0.2%)
limonene (3.7%)
p-cymenene (0.1%)
m-cymenene (0.1%)
linalool (0.6%)
cis-verbenol (0.2%)
trans-sabinol (0.5%)
pinocarvone (0.1%)

p-cymen-8-ol (1.4%)
myrtenal (0.3%)
α-terpineol (0.4%)
myrtenol (0.5%)
trans-carveol (0.3%)
cis-carveol (0.2%)
carvone (0.4%)
piperitone (0.1%)
verbenone (2.0%)
piperitenone oxide (0–2%)
δ-elemene (1.6%)
6-hydroxy-piperitol (0.6%)
α-ylangene (0.1%)
α-copaene (3.1%)
8-hydroxycarvone (0.2%)
β-elemene (0.3%)
β-caryophyllene (6.9%)
α-humulene (0.8%)
ar-curcumene (0.2%)
β-selinene (0.4%)
α-selinene (0.2%)
trans-calamenene (0.4%)
δ-cadinene (0.2%)
α-calacorene (0.2%)
isocaryophyllene oxide (1.6%)
caryophyllene oxide (29.3%)
humulene epoxide II (1.4%)
isospathulenol (3.1%)
ar-turmerone (0.7%)
caryophyllenol° (0.5%)

† incorrect identification based on GC elution order.
° correct isomer not identified

Also, trace (< 0.1%) amounts of α-thujene, o-cresol, the furanoid form of *trans*-linalool oxide, isoborneol, cryptone and m-cymen-8-ol were found in the oil.

Using SPME, Jirovetz et al. (2002) determined the headspace volatiles of an oil produced by hydrodistillation from black pepper of Cameroonian origin. The volatiles characterized in the headspace were:

acetaldehyde (0.01%)
α-pinene (6.40%)
sabinene (2.46%)
β-pinene (10.02%)
myrcene (1.38%)
α-phellandrene (8.56%)
δ-3-carene (2.78%)
β-phellandrene (0.41%)
(Z)-β-ocimene (3.19%)
p-cymene (2.14%)
limonene (10.26%)
(E)-β-ocimene (1.33%)
terpinolene (1.32%)
linalool (2.51%)
(E)-2-nonenal (1.08%)
borneol (0.21%)
p-cymen-8-ol (0.14%)
octanoic acid (0.21%)
terpinen-4-ol (1.61%)
α-terpineol (1.59%)
carveol° (0.27%)
cuminaldehyde (0.01%)
citronellol (0.11%)
carvone (0.16%)
decanol (1.04%)
nonanoic acid (0.06%)
safrole (0.52%)
cumin alcohol (0.07%)
eugenol (0.07%)
α-cubebene (0.13%)
α-copaene (1.41%)
β-elemene (2.55%)
dodecanal (0.18%)
methyl eugenol (0.92%)
α-gurjunene (0.32%)
β-caryophyllene (7.29%)
γ-elemene (0.06%)
trans-α-bergamotene (0.14%)
(E)-β-farnesene (0.41%)
α-guaiene (0.25%)
α-humulene (0.58%)
sarisan[a] (0.36%)
β-guaiene° (0.17%)
germacrene D (11.01%)
β-bisabolene (0.77%)
δ-cadinene (0.13%)
calamenene° (0.52%)
cadina-1,4-diene (0.22%)
elemol (0.16%)

Comparative percentage composition of pepper oil produced from fresh and dried peppercorns

T-1

Compound	FP-1	FP-2	DP-1	DP-2
isoamyl alcohol	0.1	-	0.3	-
amyl alcohol	0.5	-	-	-
3-hexenol*	0.5	-	-	-
octane	0.3	-	-	-
hexanol	0.5	-	-	-
4-heptanol	-	-	0.3	-
nonane	0.3	-	0.1	t
α-thujene	0.2	1.6	-	0.1
α-pinene	0.7	12.1	1.5	5.4
camphene	6.1	-	2.4	0.2
sabinene	3.0	0.3	0.9	0.2
β-pinene	2.0	14.2	1.1	15.2
myrcene	1.6	-	-	-
α-phellandrene	t	-	4.3	3.3
δ-3-carene	0.4	0.2	5.0	8.0
p-cymene	-	-	11.0	13.0
limonene	5.7	18.8	12.0	20.1
(E)-β-ocimene	-	-	1.5	-
γ-terpinene	6.3	0.3	2.0	0.2
terpinolene	4.4	-	4.2	-
cis-linalool oxide [f]	0.6	0.1	2.5	3.0
trans-sabinene hydrate	-	-	2.7	2.0
trans-linalool oxide [f]	18.6	0.2	3.3	1.0
α-thujone	1.7	-	-	-
linalool	2.8	0.3	2.3	0.5
β-thujone	1.7	-	1.7	-
myrcenol	2.0	-	2.5	-
cis-p-menth-2-en-1-ol	0.3	-	2.1	-
cis-p-menth-2-en-7-ol	0.4	-	-	-
limonene oxide*	1.0	-	2.9	3.0
trans-p-menth-2-en-1-ol	0.9	0.3	1.9	0.5
citronellal	0.3	t	1.7	1.0
menthone	1.1	0.5	-	-
(E)-2-nonenal	-	-	5.0	4.0
p-menth-8-en-1-ol	0.4	-	17.0	0.5
terpinen-4-ol	0.7	8.9	-	-
α-terpineol	25.6	0.5	2.3	0.5
dihydrocarveol	0.9	0.5	-	-
p-menth-8-en-2-ol	0.4	-	0.6	-
p-menthen-9-al*	0.9	-	-	-
nerol	0.2	-	-	-
neral	0.5	-	-	-
geraniol	0.1	-	-	-
geranial	t	-	-	-
piperitone	t	-	-	0.1
carvone oxide*	0.1	-	-	0.1
cuminyl alcohol	t	-	-	-
thymol	0.1	-	-	-
cinnamyl alcohol	t	-	0.2	-
δ-terpinyl acetate	t	-	0.2	-
α-terpinyl acetate	0.1	0.6	-	-
eugenol	-	-	t	-
geranyl acetate	-	-	t	0.2

Comparative percentage composition of pepper oil produced from fresh and dried peppercorns

T-1 (Contd.)

Compound	FP-1	FP-2	DP-1	DP-2
α-cubebene	t	0.1	0.1	1.9
α-copaene	t	1.1	1.1	1.5
β-bourbonene	t	-	-	-
β-elemene	t	-	-	0.1
α-humulene	0.2	1.9	0.1	0.4
β-caryophyllene	t	6.7	t	10.8
β-copaene	t	-	t	t
α-amorphene	t	-	0.1	1.7
α-guaiene	0.1	0.1	0.1	-
germacrene D	t	-	0.1	0.1
ar-curcumene	0.1	-	0.1	0.3
α-selinene	0.2	0.2	0.6	0.1
β-bisabolene	-	-	t	0.3
α-bisabolene*	6.4	3.7	-	2.5
zingiberene	t	-	1.0	t
γ-cadinene	t	2.6	-	2.8
(Z)-nerolidol	t	3.1	-	t
(E)-nerolidol	-	7.1	-	t
caryophyllenol*	t	-	-	0.1
caryophyllene oxide	t	0.3	-	0.4
humulene epoxide I	t	-	t	t
cedrol	0.1	-	0.1	0.1
α-eudesmol	-	-	0.1	0.1
cadina-1,4-dien-3-ol	t	-	-	-
β-bisabolene	t	-	-	-
(Z,Z)-farnesol	1.0	-	-	-
(Z,E)-farnesol	t	-	t	-

FP-1 = Fresh green pepper aroma concentrate
FP-2 = Fresh green pepper oil
DP-1 = Dried pepper aroma concentrate
DP-2 = Dried pepper oil
* correct isomer not identified
f = furanoid form
t = trace (< 0.1%)

Percentage composition of the main components of oils produced from different particle sizes of ground black pepper

T-2

Compound	1	2	3
α-thujene	1.12	2.00	0.72
α-pinene	4.59	8.58	3.38
sabinene	17.23	24.47	16.68
β-pinene	10.63	14.42	10.53
limonene	22.70	23.31	26.17
α-copaene	4.46	3.55	4.19
β-caryophyllene	2.09	1.58	1.82
β-bisabolene	7.21	4.35	6.09
caryophyllene oxide	9.88	6.82	9.58

1 = oil from 0.61 mm particle size (oil yield 2.65%)
2 = oil from 0.70 mm particle size (oil yield 2.92%)
3 = oil from 2.08 mm particle size (oil yield 2.54%)

(E)-nerolidol (1.62%)
caryophyllene alcohol (1.23%)
caryophyllene oxide (0.88%)
dodecanol† (2.21%)
spathulenol (1.05%)
β-eudesmol (1.36%)
β-bisabolol (1.26%)
(Z,E)-farnesol (1.09%)
(E,E)-farnesol (0.44%)
hexadecanol (0.12%)

† incorrect identification based on GC elution order
* correct isomer not identified
a also known as 2-methoxy-4,5-methylenedioxy-allyl-benzene

The authors also identified trace (0.01%) amounts of butyric acid, (Z)-3-hexenol, (E)-2-hexenol, 3-heptanol, α-thujene, camphene, α-terpinene, γ-terpinene, nonanal, *trans*-sabinene

Percentage composition of the oils of various cultivars of black pepper *(Piper nigrum)*

Compound	K-1	K-2	T-1	A-1	K-3	O-1	K-4	C-1
α-thujene	0-0.1	0-0.4	0.1-0.8	0.4-2.2	1.0-2.5	0.6-2.0	0.2-0.6	0.1-3.6
α-pinene	5.0-5.4	3.5-10.1	2.4-12.9	2.4-6.9	3.0-7.4	1.8-5.9	2.7-7.9	4.0-7.1
camphene	0.1-0.2	0.1-0.4	0.1-0.3	0-0.6	t-0.1	0.1	0.1-0.3	0.1-0.2
sabinene	0-0.6	1.8-7.1	3.8-4.1	5.5-20.9	11.2-22.1	0.1-26.8	1.9-5.3	9.7-22.3
β-pinene	14.1-15.2	4.4-11.7	2.0-7.3	6.1-12.5	7.5-15.4	3.8-11.7	3.8-10.9	7.7-11.2
myrcene	0-0.9	0-2.6	0.4-6.3	0-2.4	-	0-18.6	0.2-2.0	0-3.0
α-phellandrene	2.8-3.3	0.4-9.8	0.5-2.2	0.1-0.3	0.2	0.1-0.4	0.5-6.8	1.2-2.6
δ-3-carene	17.8-21.0	0.9-4.5	2.5-12.6	0.1-0.4	t-0.2	t-0.1	0-4.2	3.2-9.8
p-cymene	0.6-0.9	0-0.9	0.4-0.7	0-0.2	0.1-0.2	0.1-0.4	0.3-1.0	0.4-4.0
limonene	19.6-20.1	10.7-19.1	9.4-16.4	10.3-21.9	12.7-23.8	15.5-20.2	9.6-16.9	14.7-17.8
β-ocimene *	0-0.2	0.1-0.2	0-0.3	0.2-0.4	0.2-0.3	0.2-0.5	0-0.1	0.4-0.5
γ-terpinene	0-0.2	0-0.1	0.1-0.4	0.1-0.3	0.2-0.5	0.1-0.4	t-0.1	0.3
terpinolene	0-0.2	0-0.1	0.1	0.1-0.2	0.2-0.3	0.1-0.2	t-0.1	0.1-0.2
trans-linalool oxide [f]	0.5-1.0	0.2-0.4	0.3-0.7	0.1-0.2	0.1-0.3	0.1-0.2	0.1-0.2	0.4-0.6
linalool	0-0.5	0.7-1.3	0.6-0.6	0.1-0.3	0.1-0.5	0.1-0.3	0.7-1.2	0.3-0.6
cis-p-menth-2-en-1-ol	-	-	-	-	t-0.3	0-t	t	0-0.3
trans-p-menth-2-en-1-ol	-	0-0.1	-	-	t-0.2	0-0.1	0-t	0-t
citronellal	0-0.1	0-0.1	0-0.8	-	t-0.1	0-t	t	0-t
cis-p-menth-8-en-2-ol	0-0.2	0.1	-	-	-	0-0.1	t-0.2	0-t
borneol	-	0-0.1	-	-	0-t	-	0-0.1	0-t
terpinen-4-ol	0-0.1	-	-	-	0.5-0.9	0.3-0.8	0.1-0.3	0.3-0.5
α-terpineol	0.1-0.3	0.2-0.3	0.2	0.1-0.2	0-0.2	0.1-0.3	0-0.2	0-0.1
dihydrocarveol	0-0.1	-	-	-	-	-	0-0.3	0-0.5
trans-p-menth-8-en-2-ol	0-0.1	-	0-0.1	-	0-t	0-t	t-0.1	0-t
trans-carveol	0-0.1	0-0.1	-	-	0-t	0-t	0-0.1	0-0.1
cis-carveol	0-0.1	-	-	-	0-t	0-t	0-0.1	0-0.1
piperitone	0-0.1	0-0.1	-	0.1	0-t	t-0.1	t	0-0.1
carvone oxide *	0-0.1	-	-	-	-	0.1	t-0.2	-
myrtenol	0-0.2	0.1	-	-	-	-	-	-
α-terpinyl acetate	0.2-0.3	0.1-0.2	0.3	0.4-0.5	0.1-0.6	0.3-1.0	0.2-0.3	0.2-1.4
neryl acetate	0.1	0-0.2	0.1-0.2	0-0.2	t-0.1	t-0.1	0.1	t-0.2
geranyl acetate	0.1-0.3	0-0.1	-	-	0.1	t	0-t	0-0.1
α-cubebene	1.5-2.2	0.8-1.3	1.3-3.8	0.1-0.6	0.1-0.8	0.1-0.2	0.2-0.3	0.1-2.9
α-copaene	-	0-0.1	0.7-0.9	0.6-1.1	0.2-0.3	t-0.2	0.1-1.7	0.3-0.9
β-elemene	0-0.1	0-0.1	-	0-1.0	0.3-2.4	0-0.2	t-0.2	0.1
β-caryophyllene	19.8-25.6	25.9-47.5	23.5-45.3	24.4-46.9	8.9-24.1	20.6-33.1	29.0-46.0	17.4-23.1
trans-α-bergamotene	-	0-1.1	0-0.2	0-0.1	0.2-1.8	-	-	t-0.3
α-humulene	0-0.4	0-0.4	0-0.1	-	1.3-2.7	t-0.1	0.5-0.9	t-0.3
β-farnesene *	0-0.1	-	1.3-2.2	-	-	-	0-t	0-t
α-amorphene	1.7-1.8	1.5-3.0	1.3-2.2	1.1-2.4	0.6-1.3	1.0-1.6	1.9-3.2	1.1-1.2
α-guaiene	-	0-0.1	-	-	0-t	-	0-0.3	t
β-selinene	0.1	0.2-0.3	0.1-0.2	0.2-0.5	0.4-0.9	0.1	0.2-0.3	0.1
α-selinene	0.1	0.1-0.3	0.2-0.9	1.7-4.2	0.7-1.8	t-0.6	0.3-0.6	0.1-0.2
γ-muurolene	3.6-4.4	1.5-2.6	0.2-0.9	0.4-2.6	0.3-4.4	0.1-4.7	2.3-4.5	0.6-1.4
α-bisabolene *	2.5-2.9	2.2-3.8	-	0-1.0	0.3-5.3	t-1.9	3.0-6.0	1.1-2.3
trans-β-guaiene	-	0-0.4	0-1.7	0.1-0.4	0-2.6	0-0.2	0-0.5	0-0.1
cuparene	0.1-0.2	0.1-0.2	0-0.6	0.1-0.4	0.4-1.0	0-0.4	0.2-0.5	0.1-1.7
δ-cadinene	0-0.2	0.1	0-0.1	0.1-0.3	0.1-1.8	t-0.4	0.1-0.2	-
(E)-nerolidol	0-0.2	0-0.1	0-0.1	0.1-1.0	0.3-0.7	t-0.7	0.1	t-0.1
elemol	0.8-0.9	0-0.2	-	-	0.1-2.1	0.7-0.8	0.3	t-1.8
caryophyllenol *	0.1-0.2	0.5-0.8	-	0-0.1	0-0.6	t-0.2	t	0.1-0.3
caryophyllene oxide	0.4-0.5	1.7-4.0	0.8-3.7	0.9-3.9	0.6-1.0	0.6-2.8	1.1-1.2	0.2-0.5
cedrol	0.1	0.1-1.1	0-0.2	0.1-0.4	0.1-0.4	t-0.4	2.9-4.1	0.1-1.0
α-cadinol	0.1	0.2-0.4	0.1-0.4	0.4	0.1-0.3	0.1-0.4	0.2	0.1-0.6
T-cadinol	0.1	0-0.4	0-0.3	0.1-0.8	1.0-3.3	0.1-0.4	0.1-0.4	0.1-0.4

K-1 = 'Karimunda', K-2 = 'Kalluvally', T-1 = 'Thommankodi', A-1 = 'Arakulam munda', K-3 = 'Kottanadan', O-1 = 'Ottaplackal', K-4 = 'Kuthiravally', C-1 = 'Cheriakaniakadan', T-2 = 'Thevanmundi', V-2 = 'Vellanamban'; t = trace (<0.1%); f= furanoid form; * correct isomer not identified.

T-2	P-1	V-1	S-1	A-2	N-1	N-2	U-1	S-2	K-5	V-2
0.5-1.2	0.8-1.5	0.8-1.1	0.1	0.5-2.3	0.3-2.7	1.0-2.2	0.1-0.2	t-1.5	t-0.7	0.4-1.7
1.9-5.2	3.0-5.1	2.9-6.3	3.2-7.0	6.6-8.4	2.0-6.4	4.7-6.5	9.1-14.6	3.3-5.5	2.3-5.4	1.7-3.6
t	0.1-0.2	0.3-0.4	0.1-0.2	t-0.2	t-0.1	t-0.2	0.2-0.4	0.1-0.2	t-0.1	0.1-0.2
4.5-16.2	2.3-7.8	12.9-17.1	0.2-0.5	0-27.5	4.4-24.6	23.2-27.3	0.1-0.3	0.2-4.3	6.7-13.3	3.9-18.8
3.7-8.7	6.0-11.7	0-t	7.6-9.6	9.2-23.9	4.8-15.6	7.8-11.3	9.3-12.5	0-11.2	0-5.2	3.9-10.9
0-1.6	4.1-7.2	0.1-0.2	4.3-7.9	0-11.1	0-8.4	0-0.3	3.5-4.3	0-9.6	0-5.2	0-3.1
0.6-0.9	1.2-1.5	0.7-2.1	0.1-3.8	t-0.4	0.1-0.3	0.4-1.3	5.0-7.4	0-7.7	0-6.2	0.2-1.3
4.8-5.5	2.1-7.3	0-10.5	19.0-23.4	0-0.1	0-2.3	0.1-7.9	6.7-8.5	0.1-11.1	0.4-0.5	t-7.6
0.2-0.5	0-6.2	0-0.7	0.6-2.3	0.2-0.5	0.2-0.4	0.1-0.5	1.1-1.3	0.5-1.5	0-2.0	0.1-0.3
8.3-18.0	14.9-15.8	12.9-18.6	18.3-22.7	19.8-22.5	9.5-19.5	12.9-18.6	13.3-19.5	20.1-22.1	14.5-17.5	8.3-19.8
t-0.2	0-12.0	0-t	0-0.1	t-0.2	t-0.2	0-t	0-0.1	t-0.2	0-0.2	0-0.1
t-0.1	0.2-0.3	0.1-0.2	t-0.2	t-0.2	0.1	0.2-0.3	t-0.1	0.2	0.2-0.4	t-0.2
t-0.1	0.1	0.1	0-0.1	0.1-0.3	0.1-0.2	0.1-0.3	-	0-0.2	0.1	t-0.1
0.3	0.5	0.4-0.7	0.6-1.1	t-0.2	0.1	t-0.4	0.4-0.5	0.4-1.1	0.2-1.2	0.1-0.5
0.2-0.4	0.5-0.8	0.1	0.5-0.6	0.2-0.5	0.1-0.5	0.3-0.6	0.1	0.2-0.6	0.4	0.3-0.5
0-0.3	0-0.1	0-0.1	0-0.1	0-0.3	0-0.1	0-0.1	0-0.1	0-0.3	-	0-0.1
0-t	0-t	0-t	0-0.1	0-0.1	0-t	0-0.1	0-0.1	0-0.1	0-0.1	0-0.1
0-t	0-t	0-0.1	0-0.1	0-t	0-t	0-0.1	0-0.1	0.1	0-0.1	0-0.1
0-t	0-0.1	0-0.1	t-0.3	-	-	-	-	t-0.1	0-0.1	0-0.1
0-t	0-t	0-0.2	t-0.1	0-t	t	0-0.2	t-0.1	0-0.1	t	0-0.2
0.2-0.4	0.1-0.3	0.1-0.4	0.1-0.5	0.3-0.7	0.4-0.5	0.4-0.6	0-t	0-0.1	0.1-0.2	0.1-0.5
0.2-0.3	t-0.1	0-0.2	0.1-0.3	0.2-0.3	t-0.1	0-0.2	0.1-0.3	0.2-0.3	0.1	0-0.2
0-t	0-0.1	0-0.1	0-0.1	t-0.4	0-t	0-0.1	t-0.4	0-0.1	-	0-0.1
0-t	0-0.1	0-t	0-0.1	-	-	-	-	0-0.1	-	0-t
t-0.1	t	0-0.1	0-0.1	t-0.1	t	0-0.1	0-0.1	0.1	t	0-0.1
0-t	t-0.1	0-t	t-0.3	0-t	t-0.1	0-t	t-0.3	0-0.2	0.1	0-t
0-0.1	0-t	0-t	0-1-0.2	0-0.1	0-t	0-t	0.1-0.2	0-0.1	0-0.1	0-0.1
0-0.1	t-0.1	-	0.1-0.4	0-0.1	t-0.1	-	0.1-0.2	0-0.1	0.1	-
0-0.1	0-0.1	0-0.1	0-0.2	-	-	-	-	0-0.1	0-0.1	0-0.1
0.1-0.2	0.1-1.3	0.4-1.3	0.2-0.3	t-0.4	0.2-0.5	0.2-1.5	t	0.2	0.8-1.7	0.1-0.4
0.2-0.3	t-0.2	0.1	0.1	t	t	t-0.1	0.1-0.2	0-t	t-0.1	t-0.2
0-0.1	-	t	t	0-0.1	-	t-0.2	t-0.9	0-0.1	-	0-0.1
3.1-6.8	0.1-4.5	0.2-3.0	0.1-0.3	t	t	0.1-0.7	t-0.2	t-0.2	0.3-1.6	0.1-6.1
0.7-1.6	1.0-1.1	0.6-0.8	0.9-1.7	0.1-0.6	0.4-0.6	t-1.1	0.9-1.7	1.5-2.0	0.5-1.0	1.1-1.5
0-0.5	0.1	0-0.3	0.1-0.2	t-1.4	-	t-0.3	0-0.2	0.1-0.2	0-0.1	0.1
20.3-34.7	24.4-30.8	23.0-38.4	7.6-21.3	6.4-18.4	29.8-52.9	17.0-31.0	25.1-37.8	16.8-23.1	20.8-39.1	28.4-32.9
0-0.2	0-0.3	0-0.2	0-0.1	0-0.2	0-0.3	0-0.2	0-0.1	0-0.2	0.1	0-0.2
0.4-0.5	0-0.1	0-0.2	0.3-0.4	0.1-1.4	0-0.2	t-0.4	t-0.1	0.4-0.6	t-0.4	0-0.5
0-0.2	0-t	0-0.1	0.1-0.2	0-0.2	0-t	0-0.1	0.1-0.2	0-0.2	0-t	0-0.1
1.0-2.0	1.3-1.7	1.2-1.9	0.8-1.9	0.5-1.2	1.5-2.7	1.2-1.6	1.5-2.5	0-1.9	1.1-2.1	1.5-1.9
1.7-3.0	t-1.0	0-0.1	0-0.1	-	-	-	-	0.3-2.5	t-1.0	0-0.1
0.2-0.4	t-0.1	0.1-0.2	0.1	t-0.7	t-0.1	0.1	t-0.1	0-0.1	t-0.1	0.1-0.2
0.1-0.4	0.1-0.2	0.2-0.4	t-0.1	t-0.8	0-0.2	0-0.3	0-0.4	0-t	0.1	0.1-0.7
0.4-0.7	0.3-1.9	0.9-1.6	2.9-4.3	1.3-1.7	0.2-0.8	0.2-2.1	0.2-3.3	0-5.1	0.2-1.5	0.3-1.4
1.3-3.1	1.1-2.4	0.2-0.7	1.6-3.1	-	-	-	-	0-2.5	0.2-1.5	1.3-1.6
1.7-3.0	0-1.2	t-0.1	t	0-2.5	0-t	t-0.1	-	0-4.1	-	0-0.1
0.6-4.5	0.1-2.4	0.2-1.3	0.1-0.2	t-0.4	0.1-0.8	0.1-0.7	t-1.7	t-2.8	0.1-0.6	0.1-4.6
t-0.6	0-0.1	t-0.3	0-0.1	0-1.6	0-0.2	0-0.1	0-0.1	0-0.1	-	0-0.1
t-0.2	t-0.1	t-0.1	t	t-0.3	t-1.3	0.1-0.3	t	t-0.6	t-0.1	t-0.3
3.8-9.6	1.2-6.8	1.3-2.5	0.1-0.8	3.8-9.6	t-0.2	1.3-2.5	t-0.2	0.1-0.7	t-0.1	1.0-10.5
0.1-0.3	0.1	0-t	0.1-0.2	0.1-0.3	0.1	0-t	0.1-0.2	0.1-0.3	0.1	t-1.0
1.2-1.7	0.4-2.8	1.4-1.8	0.4-6.0	0.8-4.5	2.3-3.9	0.5-1.5	0.6-2.7	0.3-1.9	0.2-0.8	0.5-0.6
0.1-0.3	0.1-0.2	0.1	t-0.1	0.1-0.3	0.1-0.2	0.1	t-0.3	t-0.3	0-0.1	0.1-0.6
1.0-2.7	0.6-1.5	0.1-0.3	0.1-0.3	1.0-2.7	0.6-1.5	t-0.2	0.1-0.2	1.0-2.3	0.1-1.0	t-0.1
0.2-2.7	0.1-0.2	0.1-0.2	0.1-0.2	0.1	t-0.1	0.1-0.2	0.1-0.2	0.1	0.1	0.1-0.2

P-1 = 'Poonjaranmundi', V-1 = 'Valiakaniakadan', S-1 = 'Subhakara', A-2 = 'Ampiriyan', N-1 = 'Narayakodi', N-2 = 'Neelamundi', U-1 = 'Uthirankotta', S-2 = 'Sreekara', K-5 = 'Kuching', and

Average comparative percentage composition of the major headspace volatiles of microwave treated and untreated black pepper T-4		
Compound	**Treated**	**Untreated**
α-thujene	0.39–1.70	0.50–1.31
α-pinene	1.38–5.43	1.83–4.27
sabinene	5.74–14.36	5.36–11.55
β-pinene	2.76–10.90	5.12–7.55
myrcene	0.90–2.15	0.88–1.63
α-phellandrene	1.10–2.58	0.73–1.62
δ-3-carene	4.70–8.50	2.72–7.78
limonene	7.77–21.15	10.08–14.23
α-copaene	1.49–4.34	1.00–5.18
β-caryophyllene	20.99–34.26	21.01–37.28
α-cedrene	0.77–1.49	0.91–1.80
α-humulene	1.10–1.99	1.33–2.20
muurolene*	1.02–2.92	1.32–2.31
α-cadinene	0.45–1.14	0.29–1.61

* correct isomer not identified.

Average comparative percentage composition of the major headspace volatiles of microwave treated and untreated white pepper T-5		
Compound	**Treated**	**Untreated**
α-pinene	3.59–5.49	3.06–6.05
β-pinene	7.61–10.86	7.00–10.71
myrcene	2.20–3.06	2.01–3.08
α-phellandrene	2.94–4.28	2.81–4.07
δ-3-carene	15.80–23.65	14.36–20.99
p-cymene	0.51–0.87	0.46–0.83
limonene	13.48–19.71	12.66–19.75
terpinolene	0.35–1.12	0.89–1.07
α-copaene	1.06–1.82	1.11–1.73
β-caryophyllene	24.57–41.88	21.88–42.92
α-cedrene	0–0.93	0–1.09
α-humulene	1.09–1.92	1.37–2.45
α-cadinene	0.24–0.70	0.28–0.45

hydrate, isoborneol, piperitone, δ-elemene, β-cubebene, zingiberene, (E,E)-α-farnesene, elemicin, α-bisabolol, benzyl benzoate and 2-phenethyl benzoate in the same headspace.

Plessi et al. (2002) studied the effect of microwave treatment on the volatiles of both white and black pepper. Samples of white and black pepper were each ground and their headspace volatiles determined by SPME (divinyl benzene/carboxen on polydimethylsiloxane fibre). The headspace volatiles from each pepper can be seen summarized in **T-4** and **T-5**. Although there appears to be some minor quantitative differences found between treated and untreated materials, statistical analysis revealed that the samples could not be differentiated. Consequently it could be concluded that the use of microwave treatment for sanitization purposes did not affect the aromatic volatiles of white and black pepper.

Using simultaneous distillation and extraction as their method of isolation, Orav et al. (2004) examined the composition of oils produced from ground green pepper, black pepper and white pepper obtained from two separate seasons. The compounds characterized in these oils and their quantitative data are presented in summary form in **T-6**. It should be noted that the high levels of eugenol found in oils produced from ground green and white pepper is extremely unusual, so the data must be taken (no pun intended) with a grain of salt.

To obtain white pepper, ripe pepper fruits are immersed in water for a period of ca. 14 days to soften the pulpy portion from the inner pericarp, after which it is removed and the remaining pericarps are sun-dried to yield typical white peppercorns. Sometimes during this removal of the pulp the fruit undergoes fermentation reactions resulting in the formation of off-odors to the resulting white pepper which deleteriously affects the quality of the white pepper and or any oil made from such materials. Steinhaus and Schieberle (2004) used GC-olfactometry, GC/MS and/or GC/GC/MS to characterize the off-odors. They found that the most odor-active compounds in the samples of white pepper were α-pinene, linalool, 3-methylindole, heliotropin (piperonal), 4-methylphenol, myrcene, 3-methylphenol, eugenol, butyric acid, p-cymene, 2-methylbutyric acid, isovaleric acid, 3-methoxyphenol and isobutyl-3-methoxypyrazine. Of these compounds, the authors noted that 3-methylindole was responsible for the fecal, swine-manure-like odor, 3-methylphenol for the fecal, horse-like odor and the acids for the cheesy odor, although the first two of these were the main off-odors in badly fermented white pepper.

Because of the off-notes found in white pepper, Steinhaus and Schieberle (2005 a) examined the levels of 3-methylindole, 4-methylphenol, 3-methyl-phenol (smoky), butyric, 2-methylbutyric and isovaleric, valeric and hexanoic acids, (cheeselike) in samples of Muntok white pepper powder, Muntok whole white pepper, Brazilian whole white pepper and a few Indonesian whole white pepper samples from various locations in Indonesia. They found that the various samples of white pepper possessed a range of the components responsible for the off-notes, as listed below:

3-methylindole (1.0–3.8)[a]
4-methylphenol (2.6–32.0)
3-methylphenol (2.5–105.0)

butyric acid (34–155)
2-methylbutyric + isovaleric acid (2.4–8.1)
valeric acid (26–62)
hexanoic acid (29–117)

[a] mg/kg

As 3-methylindole has a low threshold, its odor activity value (concentration divided by threshold) makes it the most important contributor to the off-odor often found with white pepper. In comparison, Steinhaus and Schieberle (2005 a) found that the range of concentration of the main off-notes in samples of black peppercorns were 3-methylindole (< 0.01 mg/kg), 4-methylphenol (0.04–0.22 mg/kg), 3-methylphenol (0.09–0.33 mg/kg) and butyric acid (2.7–11.0 mg/kg).

In a follow-up paper Steinhaus and Schieberle (2005 b) proved that 3-methylindole, 4-methylphenol, 3-methylphenol and butyric acid were formed during the fermentation that took place when the fruit pulp was retted from the ripe pepper. Furthermore they described a retting process that minimized the deleterious fermentation reactions.

Clery et al. (2006) analyzed a sample of a supercritical fluid CO_2 extract of black pepper for nitrogen heterogeneous components. In this study, the following components were characterized:

pyridine (230)[a]
piperidine (150)
methylpyrazine (660)
2,6-dimethylpyridine (150)
2-ethylpyridine (150)
2,5-dimethylpyrazine (970)
2,6-dimethylpyrazine (520)
ethylpyrazine (150)
2,3-dimethylpyrazine (1.7 ppm)
2-isopropyridine (150)
2-ethyl-6-methylpyrazine (300)
2-ethyl-5-methylpyrazine (150)
2,3,5-trimethylpyrazine (1.4 ppm)
2-ethyl-3-methylpyrazine (520)
5-ethyl-2-methylpyridine (730)
2-acetylpyridine (840)
3-ethyl-2,5-dimethylpyrazine (490)
2-ethyl-3,5-dimethylpyrazine (1.5 ppm)
2,3,5,6-tetramethylpyrazine (7.0 ppm)
2-butylpyridine (390)
1-formylpyridine (28.0 ppm)
diethyl-methylpyrazine*(390)
1-acetylpiperidine (22.0 ppm)
2-heptylpyridine (400)

[a] ppb unless otherwise noted
* correct isomer not identified

Percentage composition of various oils of *Piper nigrum* **T-6**

Compound	Green pepper oil	Black pepper oil	White pepper oil
α-thujene	0.7–2.3	0.1–0.3	-
α-pinene	1.2–3.8	0.8–2.6	0.3–0.9
camphene	t–0.1	t–0.1	-
sabinene	4.5–12.2	0.8–3.8	-
β-pinene	1.8–4.7	1.5–5.9	0.7–1.7
myrcene	0.4–1.0	0.4–1.8	0.4–1.1
α-phellandrene	0.6–1.6	0.5–2.8	0.4–1.1
δ-3-carene	2.1–6.4	3.0–13.8	1.7–4.3
α-terpinene	0.2–0.3	0.1	t–0.1
p-cymene	t–0.1	0.2–0.5	0.3–0.9
β-phellandrene	1.0–2.2	t–0.1	t–0.1
limonene	3.0–7.7	3.0–14.3	2.9–6.9
γ-terpinene	0.4–1.2	0.1-0.2	-
trans-sabinene hydrate	t–0.1	t–0.1	-
terpinolene	0.2–0.5	0.1–0.8	0.2–0.3
linalool	0.4–0.8	0.5–0.6	0.5–0.7
terpinen-4-ol	1.0–1.6	0.2–0.3	-
myrtenal	t	t	t–0.2
α-terpineol	0.2–0.3	0.1	-
myrtenol	-	t	t–0.1
eugenol	1.1–18.6	1.7–2.8	2.5–41.0
α-copaene	0.2–1.9	0–4.6	t–1.5
α-cubebene	-	0.2	t–0.1
β-cubebene	-	0–3.1	-
β-elemene	0.1–1.1	0.8–1.4	t–0.2
β-caryophyllene	4.4–63.0	38.1–63.0	3.5–70.4
α-guaiene	t–1.4	0.1	t–0.2
(E)-β-farnesene	0.2–2.7	2.1–3.1	0.2–4.2
γ-muurolene	-	0.8–1.4	t–0.1
germacrene D	t–1.0	0.2	t–0.3
β-selinene	t–0.4	t–0.1	t–0.4
zingiberene	0.2–3.4	-	-
α-selinene	0.3–4.1	0.8–1.1	t–1.0
α-farnesene*	0.5–3.9	0.2	t–0.4
β-bisabolene	t–0.4	0.2–0.4	t–0.1
δ-cadinene	0.2–1.8	1.1–1.6	0.5–1.1
hedycaryol	1.3–2.2	0.1	0–1.3
(E)-nerolidol	1.4–1.8	0.1	0.2–1.4
caryophyllenol*	t–1.1	t–0.5	t–0.7
caryophyllene oxide	0.7–2.6	0.4–3.7	2.8–3.1
ledol†	0.1–0.5	t–0.1	t–0.7
β-eudesmol	t–3.6	0.6–2.7	3.5–9.7
α-muurolol	t–1.5	t–0.2	0–0.7
T-cadinol	1.1–4.6	-	t–1.5
T-muurolol	0.1–2.6	0.2–0.5	t–3.8
α-cadinol	0.1–0.4	t	0.1–1.7
α-farnesol*	0.1–0.3	t	0.1–0.6
α-farnesol*	t–1.1	t	0.1–1.7
α-bisabolol	0.8–2.7	t	t–0.8

* correct isomer not identified
† tentative identification (probably incorrect)
t = trace

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Siberian Fir Needle Oil

A survey of the early literature revealed that Kondakov (1923) identified the following components in Siberian fir (*Abies sibirica* Ledeb.) oil:

santene (5%)
α-pinene + β-pinene (25%)
camphene (15%)
α-phellandrene + limonene (6%)
bornyl acetate (40%)
bisabolone* (4%)

* correct isomer not identified

Pentegov et al. (1953) determined that the main components of Siberian fir needle oil were:

santene (1.9–3.3%)
α-pinene (18.6–29.7%)
camphene (9.4–15.8%)
δ-3-carene (4.1–9.5%)
β-phellandrene (3.6–7.3%)
terpinolene (0.6–1.4%)
borneol (1.2–3.6%)
bornyl acetate (29.4–41.5%)

Bowers and Bodenstein (1971) reported that the active component in *A. sibirica* needle and cone oils was (+)-bornyl acetate. This compound caused a sex pheromone-like activity in the American cockroach. The authors found that the (+)-isomer was 100 times more active than the (-)-isomer.

The monoterpene fraction of an oil of *A. sibirica* of Siberian origin was determined by Gornostaeva et al. (1977) to contain the following compounds:

santene (4.4%)
tricyclene (3.4%)
α-pinene (18.7%)
camphene (37.4%)
β-pinene (2.4%)
myrcene (0.4%)
δ-3-carene (12.8%)
limonene (8.3%)
β-phellandrene (4.0%)
terpinolene (0.2%)

Bornyl acetate (8.6%) was also found in this fraction.

Gornostaeva et al. (1978) determined that *A. sibirica* oil contained 0.15% phenols. The main phenols in this fraction were reported to be:

m-cresol (5.7%)
2,6-dimethylphenol (5.9%)
3,5-dimethylphenol (8.9%)
2,3-dimethylphenol (7.8%)
catechol (8.7%)
resorcinol (5.6%)
2,4,6-trimethylphenol (9.3%)
2-methylresorcinol (9.9%)

A commercial sample of Siberian fir needle oil was analyzed by Lawrence (1985) and found to contain the following components:

santene (2.1%)
tricyclene (2.8%)
α-pinene (14.1%)
camphene (22.9%)
β-pinene (2.4%)
δ-3-carene (10.1%)
myrcene (0.2%)
α-phellandrene (0.1%)
limonene (4.9%)
β-phellandrene (2.2%)
γ-terpinene (0.1%)
p-cymene (0.1%)
terpinolene (1.1%)
camphor (0.1%)
borneol (1.4%)
terpinen-4-ol (0.5%)
bornyl acetate (26.9%)
β-caryophyllene (0.8%)
α-humulene (0.4%)

A sample of Siberian pine needle oil (ex. *Abies sibirica*) was analyzed by capillary GC and ^{13}C-NMR (Kubeczka and Formacek, 2002). The constituents characterized in this oil were:

tricyclene (2.6%)
α-thujene + α-pinene (9.6%)
camphene (30.0%)
β-pinene (1.6%)
δ-3-carene (1.0%)
myrcene (1.0%)
limonene (4.4%)
p-cymene (0.5%)
camphor (0.7%)

- bornyl acetate (35.4%)
- α-humulene (0.7%)
- borneol + α-terpinyl acetate (2.5%)
- germacrene D (0.5%)
- bicyclogermacrene (0.2%)
- geraniol (0.4%)
- caryophyllene oxide (0.6%)

A trace amount (< 0.05%) of β-phellandrene was also characterized in this oil. Using chiral GC analysis, Ochocka et al. (2002) determined the enantiomeric ratios of four monoterpene hydrocarbons in *A. sibirica* oils produced commercially in Austria, Italy and Korea. The results of this analysis can be seen in **T-7**.

Comparative percentage composition of the enantiomers of four monoterpene hydrocarbons in *Abies sibirica* oil of three different origins **T-7**

Compound	Austrian oil	Italian oil	Korean oil
(1R,5R) -(+)-α-pinene	16.5	6.8	6.2
(1S,5S)-(-)-α-pinene	7.6	17.7	18.2
(1R,5R)-(+)-β-pinene	0	0.1	0.5
(1S,5S)-(-)-β-pinene	2.8	6.9	3.3
(4R)-(+)-limonene	3.0	2.5	1.2
(4S)-(-)-limonene	9.6	39.0	16.4
(3R)-(+)-camphene	1.5	1.0	2.6
(3S)-(-)-camphene	51.4	21.7	52.2

An oil produced from the fresh needles and twigs of *A. sibirica* grown in Norway was analyzed by Rohloff and Langleite (2005) using GC/MS. This oil composition was determined to be as follows:

- α-pinene (8.8%)
- camphene (17.6%)
- β-pinene (2.3%)
- δ-3-carene (9.5%)
- myrcene (0.5%)
- limonene (3.7%)
- β-phellandrene (4.5%)
- bornyl acetate (33.0%)
- borneol (6.6%)

A commercial oil of Siberian fir needle was the subject of analysis by Jirovetz et al. (2007). Using a combination of GC-FID and GC/MS the components characterized in this oil were:

- santene (1.6%)
- tricyclene (2.0%)
- α-pinene (15.2%)
- camphene (22.5%)
- β-pinene (3.0%)
- myrcene (0.6%)
- α-phellandrene (0.2%)
- p-cymene (0.1%)
- limonene (5.5%)
- δ-3-carene (12.8%)
- β-phellandrene (1.8%)
- γ-terpinene (0.2%)
- terpinolene (1.4%)
- camphor (0.2%)
- isoborneol (1.0%)
- borneol (1.2%)
- terpinen-4-ol (0.1%)
- α-terpineol (0.2%)
- bornyl acetate (27.1%)
- isobornyl acetate (1.1%)
- bornyl salicylate (1.1%)
- geranyl acetate (0.2%)
- β-caryophyllene (0.9%)
- α-humulene (0.5%)
- β-bisabolene (0.1%)
- α-bisabolene* (0.1%)

* correct isomer not identified

Although δ-3-carene is a normal constituent of Siberian fir needle oil, Jirovetz et al. noted that its retention index was 1030 on a non-polar column, while it always is between 1008–1014. This was likely either a typographical or author error.

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