Progress in Essential Oils

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Calendula Oil

An oil produced from *Calendula officinalis* L. that is known in Europe as pot marigold can be found in limited quantities in commerce.

Gora et al. (1980) reported that the essential oil of *C. officinalis* contained γ-terpinene, menthone, isomenthone, β-caryophyllene, γ-cadinene, α-copaene, α-guaiene, δ-cadinene and α-muurolene, although no quantitative data was given.

The oxygenated terpenes found in *C. officinalis* were the subject of analysis by Gracza (1987). The components characterized in the oil were α-ionone, β-ionone, carvone, β-ionone epoxide, geranyl acetone, dihydroactinodiolide, oplopanone, α-cadinol, T-cadinol, caryophyllene oxide, a β-caryophyllene ketone and a sesquiterpene lactone known as peduculatine.

Marczal et al. (1987) characterized the presence of γ-muurolene, δ-cadinene, α-muurolol, α-cadinol and a tentative identification of guaiol in an oil of *C. officinalis* of Hungarian origin.

An oil of *C. officinalis* was screened for its antimicrobial properties by Janssen (1989). The fresh flower oil used in this study was found to possess the following composition:

α-pinene (6.07%)
α-thujene (11.20%)
toluene (0.07%)
camphene (0.05%)
β-pinene (0.26%)
sabinene (0.53%)
m-xylene (0.08%)
δ-3-carene (0.11%)
myrcene (0.66%)
α-phellandrene (0.11%)
α-terpinene (0.08%)
1,8-cineole, limonene + β-phellandrene (0.85%)
(Z)-β-ocimene (0.07%)
γ-terpinene (0.16%)
(E)-β-ocimene (0.22%)
p-cymene (1.29%)
terpinolene (0.08%)
α-cubebene (0.06%)
α-copaene (0.22%)
α-gurjunene (0.07%)
β-caryophyllene (0.39%)
β-elemene (0.05%)
α-humulene (0.66%)
germacrene D (0.44%)
γ-cadinene (2.09%)
calamenene* (0.39%)
δ-cadinene (7.51%)
linalool (0.21%)
trans-p-menth-2-en-1-ol (0.07%)
cis-p-menth-2-en-1-ol (0.05%)
terpinen-4-ol (1.97%)
α-terpineol (0.33%)
cubenol (0.59%)
T-cadinol (12.71%)
α-cadinol (27.73%)
palustrol (0.46%)
β-eudesmol (1.71%)

*correct isomer not identified

Trace amounts (< 0.01%) of tricyclene, β-bourbonene, δ-2-carene, 6-methyl-5-hepten-2-one, 2-heptanone and 2-nonanone also were found in this oil. However, based on the previous data, only 79.6% of the total oil was characterized.

Chalchat et al. (1991) compared the composition of oils produced from both whole plants (in flower) and flowers of *C. officinalis* grown in France. The two oils also were compared with an alcohol extract of whole plants. The comparative oil study is presented in T-1.

Percentage composition of whole plant and flower oils of *Calendula officinalis* **T-1**

Compound	Whole plant oil	Flower oil
2-butanone	t	0.20
2-pentanone	t	0.04
α-pinene	3.64	0.20
β-fenchene	8.52	0.65
camphene	0.07	0.02
β-pinene	0.33	0.24
sabinene	0.63	0.11
δ-3-carene	0.09	0.21
α-phellandrene	0.16	0.02
myrcene	0.54	0.17
α-terpinene	0.20	0.12
limonene	0.48	1.17
1,8-cineole	0.11	–
β-phellandrene	0.09	0.25
γ-terpinene	0.65	0.37
(E)-β-ocimene	0.26	0.40
p-cymene	0.20	0.59
terpinolene	0.11	0.09
menthone	t	0.05
β-ylangene	0.10	0.13
α-gurjunene	–	0.58
β-cubebene	0.12	0.11
bornyl acetate	t	1.37
aromadendrene	0.28	2.25
β-caryophyllene	0.49	0.82
terpinen-4-ol	0.65	0.04
γ-cadinene isomers*	0.52	0.20
α-humulene	0.56	0.94
γ-amorphene	0.11	0.10
β-farnesene*	0.05	0.22
γ-muurolene	1.42	0.45
α-patchoulene	1.36	2.93
γ-cadinene	0.21	0.31
γ-patchoulene	0.07	–
α-muurolene	2.87	1.54
γ_2-cadinene	–	0.50
δ-cadinene	17.43	5.30
α-cadinene	1.03	0.46
calamenene*	0.10	0.10
calacorene*	0.15	–
nerolidol*	3.04	8.14
cubenol	0.90	0.59
epi-cubenol	0.88	7.26
bisabolol oxide*	t	0.12
T-cadinol	5.22	3.21
α-muurolol	6.97	5.67
T-muurolol	1.69	1.06
eudesmol*	0.30	7.83
α-cadinol	24.76	20.09

t = trace (< 0.01%); *correct isomer not identified

In addition, trace amounts (< 0.01%) of (Z)-β-ocimene and camphor also were found in the whole plant oil. As can be seen from the compositional data, the oils differed because the whole plant oil was richer in monoterpene and sesquiterpene hydrocarbons than the flower oil. Also, the yield of whole plant oil was 0.3%, which was higher than that of the flower oil. Finally, the composition of the ethanolic extract was determined to be as follows:

β-ylangene (0.37%)
camphor (0.03%)
α-gurjunene (0.26%)
β-cubebene (0.10%)
bornyl acetate (0.62%)
aromadendrene (1.24%)
β-caryophyllene (3.24%)
terpinen-4-ol (0.88%)
γ-cadinene isomers* (0.49%)
α-humulene (0.64%)
β-farnesene* (0.19%)
γ-muurolene (2.42%)
α-patchoulene (4.71%)
α-muurolene (1.75%)
δ_2-cadinene (1.16%)
δ-cadinene (2.87%)
α-cadinene (0.68%)
calemenene* (0.34%)
calacorene* (0.31%)
nerolidol* (2.47%)
cubenol (0.52%)
epi-cubenol (3.87%)
bisabolol oxide* (0.12%)
T-cadinol (2.95%)
α-muurolol (5.45%)
T-muurolol (1.03%)
eudesmol* (3.26%)
α-cadinol (22.67%)

*correct isomer not identified

The oil and flower headspace of *C. officinalis* both were analyzed by Radulescu et al. (2000). The oil was found to possess the following composition:

phenylacetaldehyde (1.47%)
2,5-dimethylcyclohexanol† (0.74%)
terpinen-4-ol (2.56%)
α-terpineol (1.04%)
(E)-α-ionone 5,6-epoxide (1.42%)
aromadendrene (0.49%)
α-muurolene (0.61%)
γ-cadinene (0.56%)
trimethyl tetra(H)benzofuranone† (1.52%)
δ-cadinene (2.44%)
guaiol (0.89%)
isoguaiol (1.90%)
ledol (0.90%)
β-oplopenone (1.18%)
di-epi-cubenol (1.36%)
T-cadinol (13.32%)
α-cadinol (4.88%)
α-muurolol (41.46%)
caryophyllenol* (1.18%)

†doubtful identification; *correct isomer not identified

In contrast, the headspace of the calendula flowers was reported to contain the following components:

butanol (2.86%)
isovaleraldehyde (0.45%)
valeraldehyde (1.08%)
2-propanol (0.54%)
acetic acid (4.93%)
3-hydroxy-2-butanone (0.15%)
furfuryl alcohol (0.31%)
α-thujene (0.45%)
α-pinene (0.48%)
5-methylfurfural (0.35%)
p-cymene (0.57%)
γ-terpinene (0.28%)
(Z)-β-ocimene (0.16%)

1-furfurylpyrrole (0.11%)
terpinen-4-ol (0.17%)
1-(5-methylfurfuryl)-pyrrole (0.12%)
α-guaiene† (0.21%)
α-cubebene (1.98%)
α-copaene (3.71%)
β-elemene (0.31%)
α-cadinene† (0.27%)
α-gurjunene (0.54%)
β-caryophyllene (0.47%)
β-cubebene† (0.74%)
aromadendrene (2.23%)
α-humulene (0.83%)
γ-muurolene (3.01%)
ledene (8.62%)
α-muurolene (5.89%)
γ-cadinene (8.55%)
δ-cadinene + cadina-1,3,5-triene (16.22%)
cadinene isomer* (2.48%)
α-calacorene (0.80%)
β-oplopenone (1.06%)
T-cadinol (1.06%)
α-eudesmol (1.20%)
cadalene (1.04%)
hexadecane (0.42%)
octadecane (0.38%)
nonadecane (0.48%)
hexadecanoic acid (0.39%)
eicosane (0.37%)
heneicosane (0.34%)

†incorrect identification based on GC elution order; *correct isomer not identified

Crabas et al. (2003) analyzed both an oil and a supercritical fluid CO_2 extract of the dried flowers of *C. officinalis*. Using GC/MS as their method of analysis, the oil was found to contain the following components:

hexanoic acid (0.1%)
6-methyl-5-hepten-2-ol (0.1%)
γ-terpinene (0.1%)
linalool (0.1%)
terpinen-4-ol (0.4%)
methyl dodecanoate (0.2%)
α-cubebene (0.2%)
α-copaene (1.0%)
β-cubebene (0.3%)
α-gurjunene (0.1%)
β-caryophyllene (0.4%)
β-gurjunene (0.2%)
γ-patchoulene (0.4%)
aromadendrene (0.1%)
cis-muurola-3,5-diene (0.5%)
geranylacetone (0.2%)
α-humulene (0.4%)
allo-aromadendrene (0.3%)
β-cadinene† (0.7%)
γ-muurolene (0.9%)
germacrene D + (E)-β-ionone (0.9%)
cis-β-guaiene (1.1%)
valencene (6.3%)
epi-cubebol (1.3%)
α-muurolene (1.8%)
γ-cadinene + cubebol (5.0%)
δ-cadinene (8.9%)
methyl dodecanoate (1.3%)
cadina-1,4-diene (0.6%)
α-cadinene (0.9%)
elemol (0.7%)
β-calacorene (0.2%)
dodecanoic acid (0.7%)
germacrene D-4-ol (0.4%)
viridiflorol (3.4%)
globulol + unknown (10.6%)

carotol (0.3%)
di-epi-cubenol (0.5%)
epi-cubenol (1.5%)
cubenol (2.9%)
T-muurolol (2.8%)
α-muurolol (0.8%)
α-cadinol (8.3%)
methyl tetradecanoate (3.2%)
methyl pentadecanoate (0.3%)
6,10,14-trimethyl-2-pentadecanone (0.2%)
methyl hexadecanoate (8.1%)
dolabradiene (0.4%)
hexadecanoic acid (2.0%)
ethyl hexadecanoate (0.1%)
methyl heptadecanoate (0.3%)
methyl linoleate (4.6%)
methyl linolenate (4.8%)
methyl octadecanoate (0.9%)
isoamyl dodecanoate (0.1%)

†incorrect identification based on GC elution order

In addition, trace amounts (< 0.1%) of tricyclene, α-pinene, 6-methyl-5-hepten-2-one, p-mentha-2,4-diene, menthone, methylsalicylate, α-terpineol, (E)-anethole, β-bourbonene, longifolene, (E)-α-ionone, α-calacorene, ethyl tetradecanoate and musk ambrette also were characterized as constituents of this same oil. Also, a supercritical fluid CO_2 extract of the same batch of dried flowers was found to contain:

α-copaene (0.6%)
γ-muurolene (0.6%)
germacrene D + (E)-β-ionone (0.3%)
valencene (0.4%)
epi-cubebol (0.7%)
α-muurolene (1.0%)
γ-cadinene + cubebol (4.0%)
δ-cadinene (3.2%)
methyl dodecanoate (1.6%)
α-cadinene (0.6%)
viridiflorol (0.6%)
T-cadinol (0.9%)
T-muurolol (0.7%)
α-cadinol (1.8%)
methyl tetradecanoate (4.6%)
oplopanone (1.3%)
methyl pentadecanoate (0.5%)
6,10,14-trimethyl-2-pentadecanone (1.2%)
methyl hexadecanoate (23.8%)
dolabradiene (0.6%)
ethyl hexadecanoate (0.5%)
methyl heptadecanoate (1.1%)
methyl linoleate (18.6%)
methyl linolenate (17.2%)
methyl octadecanoate (4.8%)
butyl hexadecanoate (0.9%)
isoamyl dodecanoate (2.5%)
methyl 11,14,17-eicosatrienoate (0.4%)
methyl eicosanoate (0.4%)

Trace amounts (< 0.1%) of hexanoic acid, β-cubebene, β-caryophyllene, β-gurjunene, α-humulene, β-selinene, α-calacorene, β-calacorene, α-muurolol, β-eudesmol, 9,2-octadienal, 9,12,17-octratrienal and methyl docosanoate also were found in this CO_2 extract.

Khalid and El-Ghorab (2006) examined the effect of cold treatment (5°–7°C) of the seeds of *C. officinalis* for seven to 21 days

prior to sowing on the oil composition of flower oils produced from plants grown from seeds receiving different pre-sowing conditions. The oils were compared with a flower oil produced from seeds with no pre-sowing treatments. They found that the highest oil content was obtained from flowers produced from seeds that had been kept at 5°C for seven days prior to sowing. A comparison between this oil and the oil produced from flowers—the seeds of which had not received any pretreatment prior to sowing—can be seen in T-2.

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V. Radulescu, C. Doneanu and T. Loloiu, *CGC investigation of chemical composition of* Calendula officinalis. Rev. Roumain. Chim., **45**, 271–275 (2000).

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Percentage composition of oils of *Calendula officinalis* **T-2**

Compound	1	2
α-pinene	0.28	0.28
β-farnesene*	0.16	0.25
α-humulene	0.23	0.22
α-patchoulene	0.20	0.23
allo-aromadendrene	0.22	0.20
γ-gurjunene	0.46	0.33
γ-muurolene	0.42	0.33
α-muurolene	0.58	0.52
γ-cadinene	8.27	6.06
δ-cadinene	23.83	19.81
patchouli alcohol†	3.68	1.08
α-calacorene	0.77	–
cis-muurol-5-en-4β-ol	0.35	0.58
β-calacorene	3.22	0.25
(E)-nerolidol	5.72	–
guaiol	0.13	0.19
β-acorenol	3.60	0.13
T-cadinol	0.84	–
T-muurolol	0.10	5.86
α-eudesmol	9.78	2.52
bulnesol	0.25	0.56
α-cadinol	32.01	57.85
7-epi-α-eudesmol	0.95	0.09
5-neocedranol	0.78	0.21
α-bisabolol	0.71	0.56
epi-α-bisabolol	0.26	0.52
pentacosane	0.82	0.52

1 = control, oil produced from flowers grown from untreated seeds; 2 = oil produced from flowers grown from seeds pretreated at 5°C for seven days prior to sowing; *correct isomer not identified; †incorrect identification based on GC elution order