

Influence of drying and extraction methods on yield and chemical composition of the essential oil of *Satureja hortensis*

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Abstract

The aerial parts of *Satureja hortensis*, cultivated in Iran (Research Station of Alborz, Karaj), were collected at the full-flowering stage and dried by three different drying methods (sun-drying, shade-drying and oven-drying at 45 °C). The essential oils of every treatment were obtained by hydro-distillation of the aerial parts. In addition, the essential oil of shade-dried sample was obtained by two other distillation methods (water- and steam-distillation and direct steam-distillation). The oils were analyzed by capillary GC and GC–MS. Statistical analysis showed no significant difference between oil yield (w/w) of the oven-dried sample (1.06%) compared to shade-dried (0.94%) and sun-dried (0.87%). The oil content of the shade-dried sample, obtained by hydro-distillation, was higher (0.94%) than that of the steam-distilled (0.27%). Twenty-three components were identified in the oil of *S. hortensis* in the different drying methods, including carvacrol (46.0–48.1%) and γ -terpinene (37.7–39.4%) as main components. Seventeen compounds were characterized in the oils of different distillation methods, including carvacrol (12.3–46%) and γ -terpinene (37.7–70.4%). Although the drying methods had no significant effect on oil composition of *S. hortensis*, the distillation changed the percentage of main components sharply (significant at 1%). The steam-distillation method produced the lowest amount of carvacrol and highest amount of γ -terpinene. The results showed that extraction by hydro-distillation gave the best results for *S. hortensis*, based on oil yield and carvacrol percentage.

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1. Introduction

Satureja hortensis is an annual, herbaceous plant belonging to the family *Labiatae*. It known as summer savory, native to southern Europe and naturalized in parts of North America. The main constituents of the essential oil of *S. hortensis* are the phenols, carvacrol and thymol, as well as *p*-cymene, β -caryophyllene, linalool and other terpenoids.

The green leaves and herbaceous parts of stems of *S. hortensis* are used fresh and dried as flavouring agents in

seasoning, stews, meat dishes, poultry, sausages and vegetables. The essential oil and oleoresin are used in the food industry. In addition, the essential oil of *S. hortensis* has been used in the perfume industry, either alone or with other essential oils (Sefidkon, Jamzad, & Mirza, 2004).

As a medicinal plant, *S. hortensis* has been traditionally used as a stimulant, stomachic, carminative, expectorant and aphrodisiac. The essential oil has demonstrated antimicrobial and antidiarrheic activity because of the phenols in the oil (Sefidkon & Jamzad, 2004).

The aim of this study was to test the effect of drying method (sunshine, shade and oven-drying at 45 °C) and

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also distillation method (hydro-distillation, water- and steam-distillation and steam-distillation) on the essential oil content and composition of *Satureja hortensis*.

A literature search was undertaken on effects of different methods of drying on essential oil content and chemical composition of the essential oil plants. The results showed that drying method had a significant effect on oil content and composition of aromatic plants (Basver, 1993; Deans & Svoboda, 1992; Karawya, E-Wakeil, Hifnawy, Ismail, & Khalifa, 1980; Raghavan, Rao, Singh, & Abraham, 1997). For example, the oil content of shade-dried Roman chamomil flowers was found to be larger (1.9% w/w) than there of sun-dried (0.4%) and oven-dried at 40 °C (0.9%). The drying method also had a significant effect on the proportion of the various components (Omidbaigi, Sefidkon, & Kazemi, 2004). It was also reported that the chemical composition, physical properties and antioxidant activities of yam flours were affected by drying methods to different extents (Chin-Lin, Wenlug, Yih-Ming, & Chin-Yin, 2003).

The effects of different distillation methods on oil content and composition of aromatic plants have also been previously reported. The rose-scented geranium (*Pelargonium* sp.), was processed by various hydro-distillation methods, which revealed that water-distillation of the herb gave a higher oil yield (0.16–0.22%) than water-steam distillation (0.09–0.12%) on steam-distillation methods (0.06–0.18%). The distillation methods also had effects on the percentage of oil components (Kiran, Babu, & Kaul, 2005).

The effects of distillation method and stage of plant growth on the essential oil content and composition of *Thymus kotschyanus* were also previously studied. The highest oil yield was obtained by the hydro-distillation method and the lowest by steam-distillation. The oil yield, related to distillation method and stage of plant growth, was 0.28–1.80% w/w (the highest for the complete flowering stage by the hydro-distillation method) (Sefidkon, Dabiri, & Rahimi-Bidgoly, 1999).

2. Materials and methods

2.1. Plant material

The aerial parts of *Satureja hortensis* were collected from the Research Station of Alborz (Karaj, near Tehran), at full flowering stage. A voucher specimen has been deposited in the national herbarium of Iran (TARI).

To study the effect of drying method, three methods of drying, (sun-drying, shade-drying and oven-drying at 45 °C) were investigated. In the case of sun- and shade-drying, 0.5 kg – fresh aerial parts was spread over 1 m² of area.

Table 1

Oil yields of *Satureja hortensis* by different methods of distillation from shade-dried samples

Distillation method	Means of oil yield (g)
Hydro-distillation	0.93a
Water- and steam-distillation	0.69a
Steam-distillation	0.27b

2.2. Isolation procedure

Dried aerial parts of every treatment (40 g, three replications) were subjected to hydro-distillation for 3 h, using an all glass Clevenger-type apparatus, according to the method recommended by the European Pharmacopoeia (1983), to produce oils in yields presented in Table 1.

For investigation of the effect of distillation method on oil content And composition of *S. hortensis*, two other methods of distillation, water- and steam-distillation and direct steam-distillation, were used for the shade-dried sample. The oil yields are presented in Table 1. The oils were dried over anhydrous sodium sulfate and stored in sealed vials at low temperature (2 °C) before analysis.

2.3. Gas chromatography

GC analyses were performed, using a Shimadzu GC-9A gas chromatograph equipped with a DB-5 fused silica column (30 m × 0.25 mm i.d., film thickness 0.25 µm). Oven temperature was held at 50 °C for 5 min and then programmed to 240 °C at a rate of 3 °C/min. Detector (FID) temperature was 265 °C and injector temperature was 250 °C; helium was used as carrier gas with a linear velocity of 32 cm/s. The percentages of compounds were calculated by the area normalization method, without considering response factors.

2.4. Gas chromatography–mass spectroscopy

GC–MS analyses were carried out on a Varian 3400 GC–MS system equipped with a DB-5 fused silica column (30 m × 0.25 mm i.d., film thickness 0.25 µm); oven temperature was 50–240 °C at a rate of 4 °C/min, transfer line temperature 260 °C, carrier gas helium with a linear velocity of 31.5 cm/s, split ratio 1:60, ionization energy 70 eV, scan time 1 s, mass range 40–300 a.m.u.

2.5. Identification of components

The components of the oils were identified by comparison of their mass spectra with those of a computer

library or with authentic compounds and confirmed by comparison of their retention indices, either with those of authentic compounds or with data published in the literature (Adams, 1995; Davies, 1990; Shibamoto, 1987). Mass spectra from the literature were also compared (Adams, 1995; Stenhagen, Abrahamson, & McLaffery, 1974). The retention indices were calculated for all volatile constituents, using a homologous series of *n*-alkanes.

2.6. Statistical analysis

The data were compared with SAS software by using Duncan's test.

3. Results and discussion

The oil isolated by different methods of distillation from the aerial parts of *S. hortensis*, dried under different conditions were found to be yellow liquids in yields shown in Tables 1 and 2. The analysis of variance for different distillation and drying methods are also shown in Tables 3 and 4.

The analysis of variance (Table 3) showed that the distillation method had a significant effect on the content of *Satureja hortensis* essential oil at $\alpha = 1\%$. The

Table 2
Oil yields of *Satureja hortensis* by different methods of drying obtained by hydro-distillation

Drying method	Means of oil yield (g)
Oven-drying	1.06a
Sun-drying	0.87a
Shade-drying	0.93a

Table 3
Analysis of variance for distillation methods

Source of variation	DF	MS
Distillation method	2	0.3378*
Error	6	0.0091
Total	8	–

CV = 15.1.

* Significant at 1%.

Table 4
Analysis of variance for drying methods

Source of variation	DF	MS
Drying method	2	0.0281 ^a
Error	6	0.0189
Total	8	–

^a Not significant.

highest oil yield was obtained by hydro-distillation and the lowest by steam-distillation. This may be due to this fact that, in the steam-distillation method, the situation of plant material, such as type of plant material, mode of comminution, mode of charging and grade of insulation are much more important than the other distillation methods. These results are in agreement with previous work about the effect of distillation methods on oil content and composition of other essential oil-bearing plants (Kiran et al., 2005; Sefidkon et al., 1999). There are no significant differences between oil yields obtained by hydro-distillation (0.93%) and water and steam-distillation (0.69%), so they are placed in one group.

The different drying methods had no significant effect on oil yield of *Satureja hortensis* (Table 4). Of course, plant materials dried in an oven at 45 °C had higher essential oil content (1.06% ww) than had shade-dried (0.93%) or sun-dried (0.87%) samples.

Twenty-three components were identified in the essential oil of *S. hortensis* by different drying methods, that represented 98.1–99.9% of the oils. The chemical composition of the oils can be seen in Table 5. The components are listed in order of their elution on the DB-5 column.

By hydro-distillation, the main components of the oil of shade-dried aerial parts were carvacrol (46.0%), γ -terpinene (37.7%), *p*-cymene (4.2%) and α -terpinene (3.1%). The major components of the oil of sun-dried aerial parts were carvacrol (46.8%), γ -terpinene (39.4%), *p*-cymene (4.4%) and α -terpinene (3.3%). The main components of the oil of oven-dried aerial parts at 45 °C were carvacrol (48.1%), γ -terpinene (38.4%), *p*-cymene (3.5%) and α -terpinene (3.4%).

The drying method caused some variation of the relative proportions of the components. The major compounds, carvacrol and γ -terpinene, showed no sharp difference among the three drying methods, but a higher amount of carvacrol was obtained by oven-drying. This result is in agreement with results of studies on other aromatic plants (Omidbaigi et al., 2004; Rao, Singh, Rahavan, & Abraham, 1998; Sankat & Maharaj, 1994; Venskutonis, 1997).

Eighteen components were identified in the essential oil of *S. hortensis* (shade-dried) by different distillation methods, that represented 98.1–100% of the oils. The chemical composition of the oils can be seen in Table 6. The components are listed in order of their elution on the DB-5 column.

The main components of the oil extracted by water and steam-distillation were carvacrol (44.0%), γ -terpinene (41.8%), *p*-cymene (4.3%) and α -terpinene (3.4%).

The major constituents of the oil obtained by steam-distillation were γ -terpinene (70.4%), carvacrol (12.3%), *p*-cymene (6.5%) and α -terpinene (5.8%).

Comparison of the results shows that the different drying methods had a significant effect on the percentage of main components.

Table 5
Comparison of essential oil components of *Satureja hortensis* using different drying methods (by hydro-distillation)

No.	Compound	RI ^a	Oven-drying (%)	Sun-drying (%)	Shade-drying (%)	Methods of identification
1	α -Thujene	931	1.0	1.0	0.9	RI, MS
2	α -Pinene	939	0.5	0.5	0.5	RI, MS, Col
3	Camphene	951	0.2	–	–	RI, MS, Col
4	β -Pinene	984	0.2	0.4	0.3	RI, MS, Col
5	Myrcene	999	1.9	1.9	1.8	RI, MS, Col
6	2-Octanol	1002	0.1	–	0.2	RI, MS
7	α -Phellandrene	1006	0.2	–	–	RI, MS
8	δ -3-Carene	1011	0.1	0.3	0.1	RI, MS
9	α -Terpinene	1021	3.4	3.3	3.1	RI, MS, Col
10	<i>p</i> -Cymene	1027	3.5	4.4	4.2	RI, MS, Col
11	Limonene	1033	0.5	0.4	0.5	RI, MS, Col
12	(Z)- β -ocimene	1049	0.2	0.03	0.1	RI, MS
13	(E)- β -ocimene	1053	–	0.06	–	RI, MS
14	γ -Terpinene	1059	38.4	39.4	37.7	RI, MS, Col
15	Linalool	1104	0.1	0.1	1.6	RI, MS
16	Methyl thymol	1242	0.2	0.1	0.1	RI, MS
17	Carvacrol	1301	48.1	46.8	46.0	RI, MS, Col
18	Carvacrol acetate	1371	0.2	0.03	–	RI, MS
19	β -Caryophyllene	1413	0.2	0.2	0.3	RI, MS, Col
20	Bicyclogermacrene	1488	0.3	0.3	0.1	RI, MS
21	β -Bisabolene	1504	0.6	0.5	0.6	RI, MS
22	Isoelemicine	1568	–	0.1	–	RI, MS
23	Hexadecane	1615	–	0.04	–	RI, MS
	Total	–	99.9	99.9	98.1	–

MS, mass spectroscopy; Col, co-injection; *t*, less than 0.05%.

^a RI, retention indices in elution order from DB-5 column.

Table 6
Comparison of essential oil components of *Satureja hortensis* using different distillation methods (shade-drying)

No.	Compound	RI ^a	Hydro-distillation (%)	Water and steam-distillation (%)	Steam-distillation (%)
1	α -Thujene	927	0.9	0.7	0.4
2	α -Pinene	940	0.5	0.4	1.3
3	β -Pinene	984	0.3	0.3	–
4	Myrcene	1000	1.8	1.9	3.3
5	α -Phellandrene	1008	–	0.3	–
6	2-Octanol	1010	0.2	–	–
7	δ -3-Carene	1016	0.1	0.1	–
8	α -Terpinene	1019	3.1	3.4	5.8
9	<i>p</i> -Cymene	1028	4.2	4.3	6.5
10	Limonene	1033	0.5	0.5	–
11	(Z)- β -ocimene	1036	0.1	–	–
12	γ -Terpinene	1057	37.7	41.8	70.4
13	Linalool	1103	1.6	–	–
14	Methyl thymol	1242	0.1	0.1	–
15	Carvacrol	1301	46.0	44.0	12.3
16	β -Caryophyllene	1413	0.3	0.5	–
17	Bicyclogermacrene	1489	0.1	0.5	–
18	β -Bisabolene	1504	0.6	1.2	–
	Total	–	98.10	99.90	100

^a RI is the retention indices in elution order from DB-5 column.

The results showed that by hydro-distillation a higher amount of carvacrol (46.0%) was obtained, while, by steam-distillation, the lowest amount of carvacrol (12.3%) and the largest amount of γ -terpinene (70.4%) were obtained.

Finally, it could be concluded that drying of aerial parts of *Satureja hortensis* in the oven at 45 °C and extraction of their essential oil by hydro-distillation is most suitable and is recommended for fast drying, and high-oil yield, as well as, for a high-percentage of carvacrol.

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