Effect of drying on flavour quality of Indian spearmint (*Mentha spicata* L.)

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ABSTRACT

The essential oils obtained from Indian spearmint (*Mentha spicata* L.) subjected to different drying procedures were analysed employing gas chromatography and gas chromatography-mass spectrometry techniques. Forty three flavour components were identified, of which eucarvone, cubenol and α-cadinol are reported for the first time. The essential oil had a higher percentage of limonene (26.82%) compared to Italian and American oils. Carvone to limonene ratios were 2.2:1.0 and 2.3:1.0 in the oil from fresh and shade-dried spearmint, respectively. Blanching leaves prior to drying yielded products which were unattractive with respect to colour and appearance and were also bland and odourless due to loss of volatile oil during drying. Storing fresh leaves for 12 h after spraying with water increased the volatile oil content by about 6%. Shade drying leaves resulted in a product with a good green colour and minimum loss of volatile oil compared to other drying methods.

Key words : drying, essential oil, flavour quality, *Mentha spicata*, spearmint.

Introduction

The essential oil of Indian spearmint (*Mentha spicata* L.), a culinary herb has been the subject of study by several researchers. Karawya, Hifnawy & El-Hawary (1977) studied the composition of Egyptian oil of *M. spicata* of different maturities. Nagasawa *et al.* (1974) and Torres & Retamar (1975) reported (-) carvone as the major compound (> 60%) present in spearmint oil of Japanese and Argentinian origin, respectively, besides several other flavour components. However, the former described *cis* carvyl acetate (1.8%) as the major compound responsible for the characteristic odour associated with the Japanese oil. Detailed studies were reported on the botanical variations in different chemotypes (Kokkini & Vokou 1989),

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volatile monoterpenoid content of plantlets produced by shoot tip culture and accumulated in glandular trichomes (Gershenzon, Maffei & Croteau 1989; Hirata *et al.* 1990), the essential oil composition of different clones (Tucker & Fairbrothers 1991) and characterization of a spearmint mutant that resembles peppermint in monoterpene content (Corteau *et al.* 1991). Shalaby *et al.* (1988) observed that carvone content decreased in spearmint oil stored up to 1 year irrespective of the storage temperature. Hazra, Kahol & Ahmed (1990) reported that the yield of oil increased in the first 48 h of drying of *M. arvensis* and then steadily declined with further drying. However, there is no report on the changes in flavour composition of spearmint during drying. This paper presents the data on the effect of spearmint drying by different procedures and a comparative evaluation of flavour composition of essential oils of fresh and dried herb.

Materials and methods

Fresh spearmint was procured from the local market and the leaves were subjected to different drying procedures either as such or after blanching.

Convection drying

Fresh leaves (1 kg) were washed in running water and excess water drained. The leaves were then spread on four wire mesh trays (50 cm × 50 cm), placed in a convection dryer (Scientific Instruments Co., India) and dried at 45°C. Samples were drawn after 4 h and after completion of drying (6 h). In another experiment, fresh leaves (500 g) were initially blanched by placing in a wire mesh basket, dipping in boiling water (1min) and draining off the water. The blanched leaves were spread on two wire mesh trays and dried in the convection dryer at 45°C. The moisture and volatile oil content in fresh and blanched leaves as well as in samples drawn at intermediate stages of drying and in the final samples were determined as per standard procedures (ASTA 1985).

Cross-flow drying

One kg of fresh spearmint leaves were throughly washed in water. The leaves were distributed in three stainless steel trays (80 cm × 40 cm) and dried in a cross-flow dryer (Armstrong Smith Pvt. Ltd., India) at a temperature of 40°C after draining off the water. Samples were drawn after 4 h and after completion of drying (6 h). The experiment was repeated for the blanched herb (600 g) by spreading it evenly on two stainless steel trays and collecting the dried material at the end of 8 h drying. The moisture and volatile oil contents were determined in different samples.

Sun drying

Fresh washed spearmint leaves (700g) were sun dried (5 h) by spreading evenly on a raised platform (100 cm × 60 cm) exposed directly to sunlight. The leaves were raked once an hour to enable uniform drying. The moisture and volatile oil contents were determined in the fresh and dried samples.

Shade drying

This was carried out by two methods. In the first method, 700 g of spearmint leaves were spread inside a room over an area of 100 cm × 60 cm and allowed to dry for 24 h at room temperature (25 ± 2°C). Periodic raking of leaves was done to assist in uniform drying. In another experiment, two lots (200, 700 g) of fresh leaves were spread on the

ground inside the room, sprayed with water (50, 200 ml) and left for 12 h. The volatile oil content was determined using the first lot. The other portion was shade dried for a further 24 h. The moisture and volatile oil contents were determined in the fresh and dried herb.

Fresh spearmint oil

Fresh spearmint leaves (950 g) were transferred into a 10 l round bottomed flask, and 7 l water was added. The contents were subjected to distillation in a convectional Clevenger type apparatus for 6 h. The volatile oil obtained (10.5 ml) was dried over anhydrous sodium sulfate and stored in a sample vial in a refrigerator. The stem portion (600 g) was also hydrodistilled to obtain the volatile oil (traces).

Volatile oils from dried spearmint

Dried spearmint leaves (30-50 g) obtained by different drying methods were hydrodistilled, the volatile oils recovered and dried over anhydrous sodium sulfate.

Gas chromatographic analysis

The analysis of volatile oils from fresh and dried spearmint was carried out using a Shimadzu GC-9APF gas chromatograph fitted with a fused silica capillary column (30 m × 0.5 mm i d) coated with Supelcowax 10 and a flame ionisation detector. Column temperature was initially isothermal at 60°C for 6 min and then programmed at the rate of 2°C per min up to 180°C where it was held for 5 min. The injector block and detector temperatures were maintained at 150 and 200°C, respectively. Nitrogen was used as the carrier gas (1 ml/min) with hydrogen and air flow set at 30 and 300 ml per min, respectively. The volatile oil samples were diluted with acetone (1+9 v/v) and 0.5 µl injected for analysis.

Gas chromatography - Mass spectrometry analysis

A Hewlett-Packard 5995 GC-MS quadrupole mas spectrometer fitted with a fused silica capillary column (30m × 0.5 mm i d) coated with Carbowax 20M was used for analysis of fresh spearmint oil. The GC column temperature was initially isothermal at 70°C for 6 min, then programmed to 190°C with an increment of 2°C per min and held at the final temperature for 14 min. Helium was used as the carrier gas (5 ml/min). The injector block temperature was 200°C while the ion source temperature 180°C.

Results and discussion

Volatile oil content

The volatile oil content of leaves ranged from 0.82 to 1.53% in different samples of spearmint. Loss of volatile oil during drying was 45 and 33%, respectively, when convection and cross flow dryers were used. Blanching softened the leaves which became a lumpy mass and drying was difficult. Moreover, there was a heavy loss of volatile oil also. The blanched and dried samples had a dark (olive) green colour and were bland and odourless when compared to unblanched samples. Sun drying yielded a product with unattractive colour and appearance compared to mechanically dried products. Shade drying (25-27°C) resulted in a product with good green colour and higher volatile oil content. The volatile oil retention was 86.5% of the value in fresh leaves. Storage of fresh leaves with spraying of water improved volatile oil content of fresh and dried samples. This may be due to

increase in rate of release of flavour components from the precursors in presence of water. Hence it is advisable to keep freshly harvested mint leaves spread on a clean surface overnight sprayed with water before the herb is taken for drying. The overall quality of shade dried leaves was better compared to the leaves dried by the other three methods (Table 1).

Flavour quality

Gas chromatographic profiles of volatile oils obtained from fresh and dried spearmint are given in Figs. 1 & 2. The identification of the components was done using GC and GC-MS. Forty three components were identified based on mass spectral data (Table 2). Eucarvone, cubenol and α-cadinol are reported for the first time in spearmint oil. The relative concentration of 19 major components of the oil obtained from fresh, cross-flow dried, shade dried and sun dried spearmint leaves are given in Table 3. Carvone and limonene together

Table 1. Effect of blanching and drying on volatile oil content of spearmint

Drying method	Sample pretreatment	Time of drying (h)	Moisture content (%)	Volatile oil content * (%)
Convection	'As is'	0	89.6	1.20
		4	10.3	0.82
		6	7.5	0.66
	After blanching	0	88.8	0.85
		8.5	10.0	0.05
Cross-flow	'As is'	0	85.6	1.53
		4	7.9	1.05
		6	6.6	1.02
	After blanching	0	87.3	0.82
		8	10.0	0.09
Sun	'As is'	0	86.7	1.48
		5	10.5	0.66
Shade	'As is'	0	86.7	1.48
		24	10.2	1.28
Sprayed with water and kept for 12 h	'As is'	0	86.7	1.57
Sprayed with water and kept for 12 h and shade dried	'As is'	24	8.0	1.38

* Moisture free basis

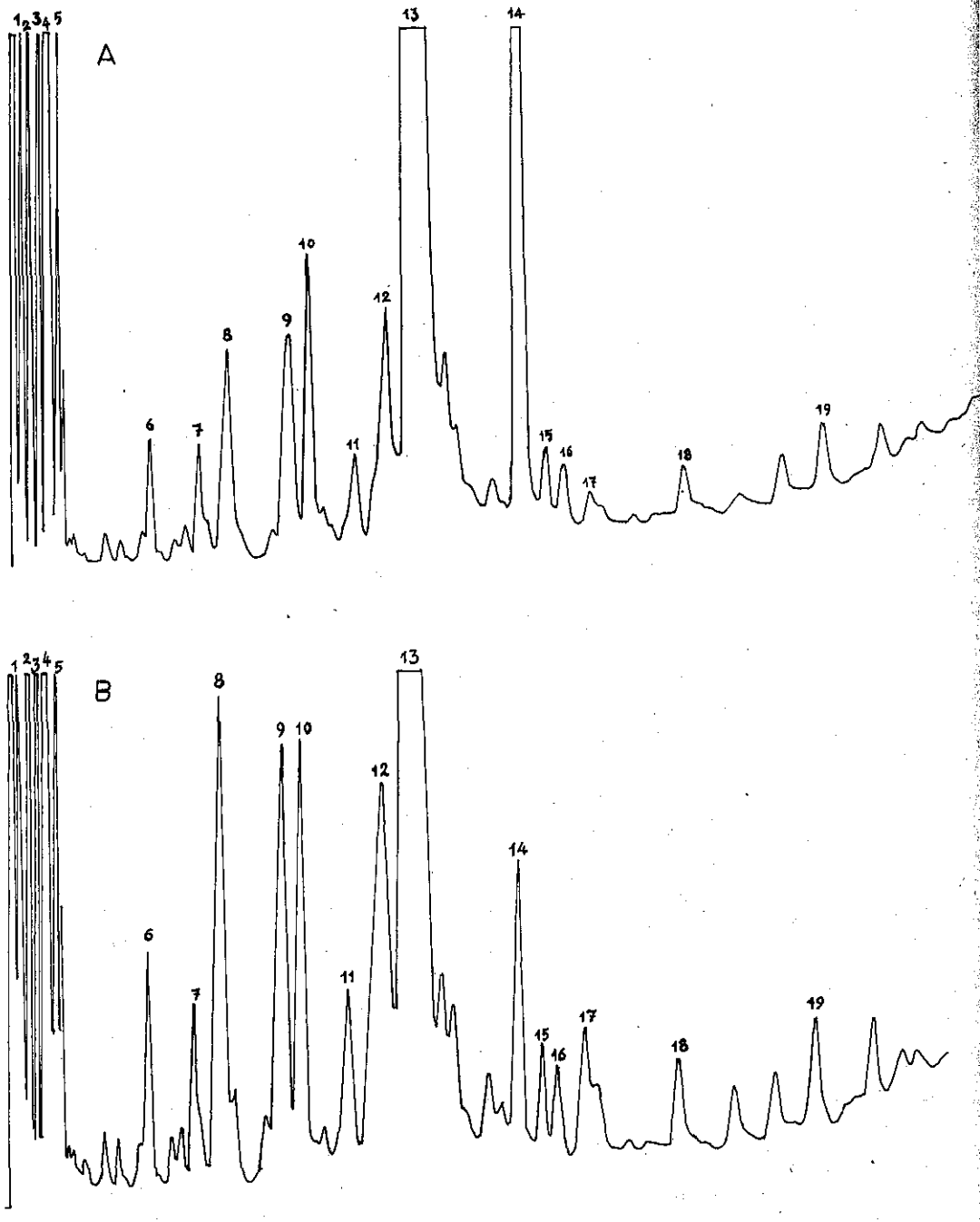

Fig. 1. Gas chromatographic profiles of essential oils obtained from (A) fresh and (B) cross-flow dried spearmint

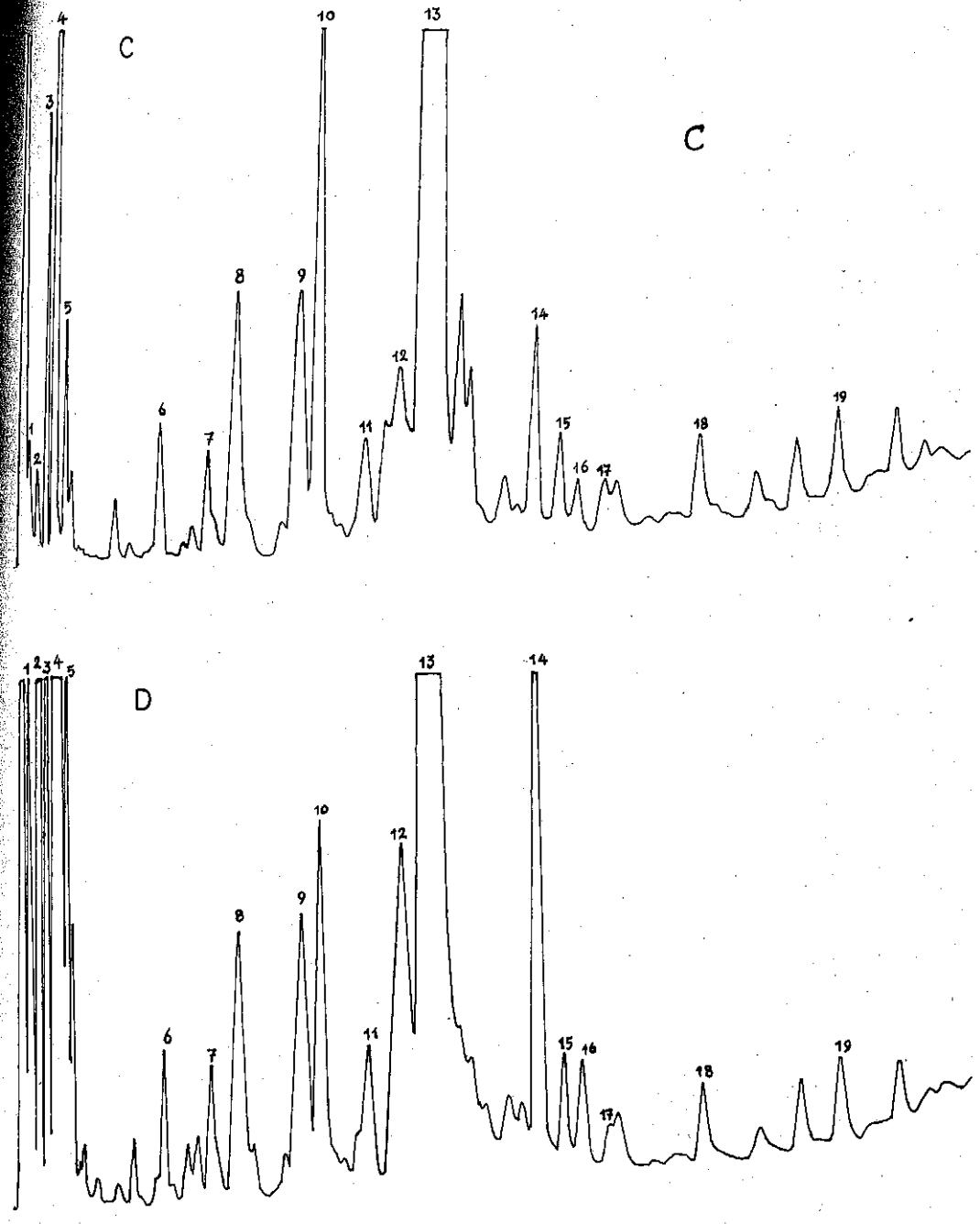

Fig. 2. Gas chromatographic profiles of essential oils obtained from (C) sun dried and (D) shade dried spearmint

Table 2. GC-MS identification of flavour constituents in fresh spearmint oil

Sl. No.	Compound	Molecular weight	Mass spectral data (m/z)
1.	α-Pinene	136	93, 77, 41, 79, 43, 53, 91, 105, 121, 136
2.	β-Pinene	136	93, 41, 77, 57, 69, 79, 43, 121, 136
3.	Myrcene	136	41, 69, 93, 53, 79
4.	Limonene	136	68, 67, 41, 93, 53, 79, 91, 77
5.	1, 8-Cineole	154	93, 41, 79, 91, 77, 53, 92
6.	*cis*-Ocimene	136	93, 41, 79, 77, 91, 53, 105, 80, 120
7.	*trans*-Ocimene	136	43, 93, 41, 79, 77, 53, 91, 67
8.	3-Octanol	130	59, 41, 55, 43, 83, 101, 57, 44
9.	*iso*-Octyl acetate	172	43, 117, 44, 132, 58, 91, 115, 65
10.	1-Octen-3-ol	128	57, 43, 41, 55, 67, 70, 72, 85
11.	Sabinen hydrate	154	43, 41, 67, 55, 44, 68, 79, 93, 110
12.	*cis*-3-Hexenyl acetate	142	41, 41, 67, 57, 82, 81, 55, 85
13.	β-Bourbonene	204	81, 43, 41, 80, 79, 123, 90
14.	α-Copaene	204	41, 43, 91, 105, 77, 119, 55, 161
15.	Linalool	154	43, 41, 55, 71, 93, 69
16.	β-Caryophyllene	204	41, 91, 79, 93, 105, 69, 43
17.	*trans*-Dihydro carvone	152	67, 41, 95, 55, 82, 68, 53, 43
18.	β-Elemene	204	41, 67, 93, 68, 79, 43, 53, 105
19.	*cis*-Dihydro carvone	152	67, 43, 41, 95, 68, 55, 44
20.	β-Cedrene	204	41, 91, 161, 105, 43, 79, 55
21.	Alloaromadendrene	204	43, 91, 41, 105, 77, 161
22.	Germacrene-D	204	41, 161, 43, 91, 77, 119, 55
23.	Dihydro carveol	154	41, 67, 79, 55, 93, 121, 107,136,
24.	*cis* β-Farnesene	204	41, 69, 93, 55, 91, 79, 105, 133, 161
25.	Borneol	150	43, 95, 41, 59, 44, 55, 67, 79, 110

26.	τ-Murrolene	204	161, 41, 91, 105, 43, 55, 77, 119
27.	Carvone	150	82, 54, 93, 108, 107, 53
28.	Valencene	204	41, 161, 105, 91, 77, 79, 81, 119
29.	τ-Cadinene	204	41, 43, 44, 91, 161, 105, 79, 77, 93
30.	δ-Cadinene	204	41, 43, 161, 119, 91, 105, 134
31.	Calamenene	202	159, 43, 129, 115, 128, 202
32.	*iso*-Dihydro carveol	154	43, 93, 107, 121, 79, 67
33.	*trans*-Carvyl acetate	194	41, 91, 109, 84, 55, 77, 53, 119, 69
34.	*trans*-Carveol	152	109, 91, 55, 41, 79, 84, 77, 93
35.	*cis*-Carveol	152	41, 43, 55, 67, 79, 93, 107, 121, 91, 69
36.	*cis*-Carvyl acetate	194	41, 43, 91, 84, 55, 79, 69, 109, 119
37.	Eucarvone	150	107, 41, 91, 150, 43, 79, 53, 51, 135
38.	*cis*-Jasmone	164	43, 41, 79, 55, 77, 91, 53, 122, 149, 164
39.	Cubenol	222	41, 43, 119, 105, 91, 161, 179, 55, 204
40.	Methyl-3-methoxy benzoate	166	43, 44, 135, 77, 92, 166, 107
41.	Eugenol	164	43, 77, 91, 164, 149, 103, 55
42.	α-Cadinol	222	43, 41, 95, 79, 105, 121, 161, 91, 204
43.	Diethyl phthalate	222	149, 43, 105, 177, 76, 65, 222

constituted more than 85% concentration in fresh oil. It is interesting to note that the ratio between these two compounds is 2.2:1.0 which contributes to the fruity/sweet character to the herb as higher limonene content is normally associated with citrus odour. However, limonene content in spearmint oils of Italian and American origin have been reported to be 5.9 and 11.4% with a carvone to limonene ratio of 6.7: 1.0 and 5.2:1.0, respectively (Maffei, Codignola & Fieschi 1986). Further, the total monoterpene hydrocarbons amount to 31.27% in fresh spearmint oil in the present study compared to 8.03% in Italian and 29.90% in American oils. The higher monoterpene hydrocarbon

Table 3. Constituents of fresh and dried spearmint oils

Peak no.*	Retention time(min)	Volatile constituent	Constituent (relative %)			
			Fresh	Cross-flow dried	Shade dried	Sun dried
1	0.992	α-Pinene	0.77	0.83	0.76	0.01
2	1.558	β-Pinene	1.04	0.87	0.86	0.03
3	2.158	Myrcene	2.84	1.42	1.53	0.30
4	2.667	Limonene	26.82	17.50	26.96	3.78
5	3.367	1, 8-Cineole	0.52	0.54	0.28	0.27
6	9.317	3-Octanol	0.43	0.38	0.16	0.31
7	12.233	1-Octen-3-ol	0.35	0.35	0.20	0.34
8	13.983	β-Bourbornene	0.40	1.27	0.50	1.13
9	17.825	Linalool	0.58	1.20	0.64	1.20
10	19.050	*trans*-Dihydro carvone	0.75	0.86	0.60	1.84
11	22.008	Germacrene-D	0.14	0.48	0.31	0.49
12	24.100	Dihydrocarveol	0.12	1.50	0.97	1.06
13	26.183	Carvone	59.59	68.39	62.78	82.90
14	32.717	*trans*-Carvyl-acetate	4.34	0.53	0.91	0.68
15	34.275	*cis*-Carvyl-acetate	0.15	0.19	0.13	0.32
16	35.350	Eucarvone	0.14	0.17	0.15	0.18
17	37.033	*cis*-Jasmone	0.12	0.46	0.15	0.19
18	42.942	Cubenol	0.20	0.26	0.17	0.42
19	51.700	α-Cadinol	0.30	0.30	0.16	0.36

*Refer Figs. 1&2

content in the oil may have a beneficial effect in mellowing down the harsh note of oxygenated terpenes. During drying all the major components were retained in the oils. There was a substantial loss of limonene in the cross-flow dried and sun dried samples. However, the total monoterpene hydrocarbon content was less in cross-flow dried and sun dried samples. These two methods may therefore be discounted for processing the herb. Interestingly, limonene was retained totally in shade dried spearmint. The derivatives of carvone constitute about 9.0% of carvone in the fresh spearmint oil while they amount to 4.5, 4.0 and 4.7% in cross-flow dried, shade dried and sun dried samples, respectively. *Trans*-carvyl acetate content in shade dried sample was about 21% of

that in the fresh material whereas carvone content had increased. These observations lead us to conclude that shade drying is the preferred method to process and preserve spearmint.

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