

Influence of Drying on the Flavor Quality of Spearmint (*Mentha spicata* L.)

M. CONSUELO DÍAZ-MAROTO,* M. SOLEDAD PÉREZ-COELLO,
 M. A. GONZÁLEZ VIÑAS, AND M. DOLORES CABEZUDO

Área de Tecnología de los Alimentos, Facultad de Ciencias Químicas (UCLM),
 Campus Universitario s/n, 13071 Ciudad Real, Spain

Spearmint (*Mentha spicata* L.) was dried using three different drying methods: oven-drying at 45 °C, air-drying at ambient temperature, and freeze-drying. The effect of the drying method on the volatile compounds and on the structural integrity and sensory characteristics of the spice was evaluated. The volatile components from fresh and dried spearmint samples were isolated by simultaneous distillation–extraction (SDE) and analyzed by gas chromatography–mass spectrometry (GC-MS). A total of 28 compounds were identified, carvone, limonene, and 1,8-cineole, in that order, being the main components in all of the samples. Oven-drying at 45 °C and air-drying at ambient temperature were the methods that produced the best results. An increase in monoterpenes was observed in all of the dried samples, except in the freeze-dried samples that underwent freezing at –198 °C. Freeze-drying resulted in substantial losses in oxygenated terpenes and sesquiterpenes. The effect of each drying method on leaf structure was observed by scanning electron microscopy. From a sensory standpoint, drying the spearmint brought about a decrease in herbaceous and floral notes together with an increase in minty odor.

KEYWORDS: *Mentha spicata* L.; spearmint; drying; volatile compounds

INTRODUCTION

The use of spices has increased significantly over the past few years, due partly to renewed interest in dishes that use a wide variety of spices and to the ability of spices to act as antioxidants in addition to their seasoning properties (1).

Mints are regarded as one of the most important spices throughout the world. The essential oils of mints are widely used as flavorings in the food, cosmetic, and pharmaceutical industries. The mints most commonly used in food are peppermint (*Mentha piperita* L.), cornmint (*Mentha arvensis* L.), and spearmint (*Mentha spicata* L.).

The chemical composition of the essential oils in spearmint has been studied by different researchers. Carvone is the major component in all cases and is the character-impact component in spearmint, followed by limonene and 1,8-cineole (2–4).

Spices can be marketed fresh or dried. The drying of spearmint is an effective method that increases the shelf life of the final product by slowing the growth of microorganisms and preventing certain biochemical reactions that may alter the organoleptic characteristics. However, drying causes changes in the product, mainly associated with fragrance and appearance, although the exact chemical nature of these changes is not clear.

The effect of a particular drying method on the release or retention of volatile compounds is not predictable and depends on the compound and the spice concerned. Oven-drying and freeze-drying of dill lead to decreases in most of the volatile compounds compared with the levels in the fresh spice (5, 6). The same occurs in parsley (7). In contrast, the effect of oven-drying at 30 °C and freeze-drying on the volatile compounds in thyme and sage has been minor, whereas losses at 60 °C were 43% in thyme and 31% in sage (8).

Microwave-drying produced greater losses in volatile compounds than oven-drying in rosemary, although it did preserve the spice's characteristic green color (9). Likewise, freeze-drying preserves the characteristic appearance of the fresh product (10), although causing substantial losses to certain volatiles in the cases of parsley and bay leaf (7, 11), whereas shade-drying of spearmint leaves has resulted in a product with a good green color and few losses of volatiles (12).

On the other hand, certain compounds normally present have been observed to increase in different spices after drying, for example, eugenol in bay leaf (11), thymol in thyme (8), and some sesquiterpenes in different spices (6, 13–16).

The present study has examined the influence of three different drying methods, oven-drying at 45 °C, air-drying at ambient temperature, and freeze-drying, on the volatile compounds in spearmint, as well as the effect of the drying method on cell structure and on the sensory characteristics of the final product.

* Author to whom correspondence should be addressed [e-mail: mc Diaz@gata-cr.uclm.es; telephone: 00 34 926 295300 (ext. 3425); fax: 00 34 926 295318].

MATERIALS AND METHODS

Samples. Fresh leaves of cultivated *Mentha spicata* L. were collected in the province of Ciudad Real (Spain) in the month of June. The sample was divided into five batches. One batch was set aside at 5 °C and analyzed before 24 h. The remaining four batches were immediately dried using one of the drying methods tested. The initial moisture content of the plant material was 76.6% dry weight. Four replications of each treatment were performed.

Drying Methods. One sample of fresh spearmint was air-dried at ambient temperature in a dark, well-ventilated room for 5 days (mean temperature = 28 °C; mean relative humidity = 40%). The final moisture content was 10%. A second sample of fresh spearmint was oven-dried using a laboratory oven at 45 °C. After 12 h, the moisture content of the spice was 8.3%. The last two samples were frozen at -18 and -198 °C, respectively. The frozen material was then freeze-dried under vacuum (1.1×10^{-2} mB). Condenser temperature was -53.2 °C. The dried material had moisture contents of 5.7 and 7.8%, respectively.

The drying conditions employed in each of these methods were selected after trials had been conducted to achieve a percentage moisture content of <10% using the lowest temperature and shortest possible time. The moisture content of the dried samples was determined (in duplicate) using a laboratory oven at 105 °C.

Extraction and Concentration of Volatiles. A microscale simultaneous distillation-extraction apparatus (Chrompack, Middelburg, The Netherlands) was used as previously described (17). An amount of 1 g of spearmint leaves in 60 mL of water with 150 μ L of ethyl myristate added as internal standard was extracted under atmospheric conditions for 2 h using dichloromethane as the extraction solvent. The extracts obtained were stored frozen at -18 °C for gas chromatographic analysis. Four replications of each extraction were performed.

Analysis of Volatiles. A Hewlett-Packard G 1800 B GCD System equipped with a gas chromatograph and a quadrupole mass detector (Hewlett-Packard, Palo Alto, CA) was used. An amount of 1 μ L of extract was injected in splitless mode (split ratio of 1:20) on an SPB-1 (Supelco) methyl silicone column measuring 50 m \times 0.25 mm, with a film thickness of 0.25 μ m. The column temperature program was 70° C (3 min), raised at 4° C/min to 120 °C, and then raised at 8 °C/min to 250 °C. Injector temperature was 250 °C. Transfer line temperature was 280 °C. Mass detector conditions were as follows: electronic impact (EI) mode at 70 eV; source temperature, 178 °C; scanning rate, 1 scan/s; mass acquisition range, 35–350.

Identification of the oil constituents was based on comparison of their GC retention indices and mass spectra with authentic standards [α -pinene, 1,8-cineole, limonene, borneol, *R*-(-)-carvone, β -caryophyllene] from Sigma-Aldrich. The tentative identification of compounds for which it was not possible to find reference volatiles was carried out by comparison of their mass spectra with spectral data from the Wiley G 1035 A library and the literature and on the basis of retention indices published in the literature (18–20). Semiquantitative analysis of the positively identified compounds was performed by the internal standard method, assuming that component response factors were the same as the response factor for the internal standard.

Scanning Electron Microscopy (SEM). Fresh and dried leaves of spearmint were examined by SEM. The leaves were broken into smaller pieces. The fragments were attached to SEM stubs and subsequently coated with gold (25 nm for 4 min) using a K-550 gold sputter coater. The coated samples were examined under a scanning electron microscope (Philips XL-30, Amsterdam, The Netherlands), and Polaroid pictures were taken.

Sensory Evaluation. Sensory evaluation was carried out on the fresh and dried samples. Free choice profiling was employed using 10 untrained assessors who furnished spontaneous information on all of the attributes (21). Each sample was placed in a glass flask (diameter = 40 mm, volume = 50 mL) sealed by a screw-cap top. Assessors sniffed the samples after they had stood for 20 min at room temperature. Attribute intensity was evaluated by each assessor using unstructured 10 cm line scales, bound at the ends by the terms “weak” and “strong”. All assessors performed three replicates.

Statistical Analysis. Principal component analysis (PCA) and the Student–Newman–Keuls test (SPSS, Program 2000) were used to assess the significance of the differences among the various treatment groups. Free choice profiling data were processed by means of Generalized Procrustes Analysis (GPA) using the Procrustes for PC program version 2.2 (Oliemans, Punter and Partners, Utrecht, The Netherlands).

RESULTS AND DISCUSSION

Table 1 sets out the mean and relative standard deviation (RSD) values for the compounds identified and quantified (micrograms per gram of dry weight) in the fresh spearmint and in the spearmint dried by the different drying methods tested. The table also summarizes the results of the Student–Newman–Keuls test for comparison of means.

Quantitatively, carvone was the most important of the 28 compounds identified, followed by limonene and 1,8-cineole. These findings are in agreement with the results obtained by other workers using supercritical fluid extraction of *Mentha spicata* subsp. *insularis* leaves grown in Italy (3) and using steam distillation of *M. spicata* leaves grown in Jalisco (4). Carvone exists naturally in two different enantiomeric forms, with *R*-(-)-carvone being the source of the typical spearmint aroma and *S*-(-)-carvone being the source of the typical caraway aroma (22). These two enantiomers are readily analyzable by direct thermal desorption coupled with chiral gas chromatography (23). In addition to the aforesaid compounds, monoterpenes were present at substantial concentrations, such as β -pinene, β -myrcene, and *trans*-thujan-4-ol. Also present were the carvone derivatives dihydrocarvone, dihydrocarveol, and carvone acetates, which may exert an effect on the overall spice aroma, because they were present in concentrations above the respective odor thresholds. Certain sesquiterpenes, such as β -bourbonene, β -caryophyllene, and *epi*-bicyclosesquiphellandrene, were also present at high concentrations.

The effect of drying on the release or retention of volatiles in spices depends on the compound and on the spice concerned (8, 10). Drying at ambient temperature and oven-drying at 45 °C resulted in increases in most of the monoterpenes and, to a lesser extent, in the carvone and certain of the carvone derivatives. These same increases were also observed in the freeze-dried samples frozen at -18 °C.

Certain of these substances may have their origin in the dehydration of oxygenated compounds, although it seems more likely that the cell damage produced by drying contributes to the release of these same substances. Biosynthesis of monoterpenes takes place in the secretory cells of the glandular trichomes, and in plants of the genus *Mentha* the monoterpenes are stored in a subcuticular compartment of the trichomes, which remains intact unless the leaves are damaged (24, 25).

Figure 1 displays scanning electron micrographs of the surface of the fresh spearmint samples and the spearmint samples dried using the different drying methods tested. The epithelial cells in the dried samples can be observed to have collapsed and split open. Some workers have also observed cell structure to collapse during drying in oregano and basil (10, 14).

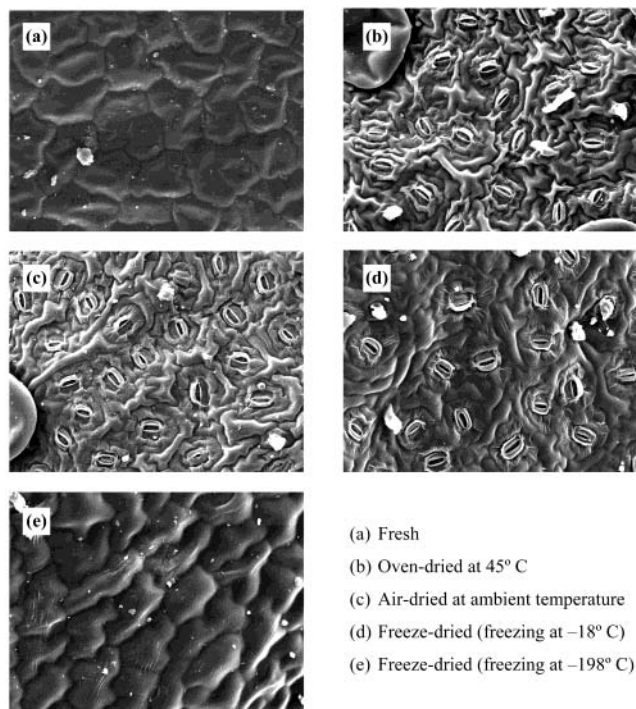
There was less cell damage in the case of freeze-drying with freezing at -18 °C, but still the damage is plain. In fact, the variation in the chemical composition of these samples was intermediate between the air-dried samples and the freeze-dried samples frozen at -198 °C.

The highest losses in volatiles occurred in the freeze-dried samples frozen at -198 °C. This was particularly true for the

Table 1. Concentration of Volatile Compounds (Micrograms per Gram of Dry Weight) in SDE Extracts of Fresh and Dried Spearmint

compound	retention index (apolar)	fresh		oven-dried at 45 °C		freeze-dried (−198 °C)		freeze-dried (−18 °C)		air-dried at ambient temp	
		mean (n = 4)	RSD (%)	mean (n = 4)	RSD (%)	mean (n = 4)	RSD (%)	mean (n = 4)	RSD (%)	mean (n = 4)	RSD (%)
α -thujene	925	10a	13.0	15b	2.9	11ac	8.5	12c	6.4	15b	8.6
α -pinene	933	307a	8.8	423b	8.0	334a	5.8	380b	4.6	407b	9.1
camphene	946	38a	6.4	53b	8.2	39a	9.8	43ac	5.4	48c	8.0
1-octen-3-ol	963	12a	14.3	11a	12.0	7b	10.8	6b	6.7	8b	10.9
sabinene	968	296a	7.2	349b	11.9	286a	9.0	333ab	6.6	364b	6.7
β -pinene	973	464a	6.7	609b	7.6	475a	6.4	545b	7.5	584b	6.4
β -myrcene	983	325a	8.2	360a	4.9	277b	7.7	320a	3.9	361a	7.3
1,8-cineole + limonene	1024	6488a	7.5	7909bd	5.0	5598c	5.1	7492b	3.6	8319d	6.6
<i>trans</i> -thujan-4-ol	1056	465a	9.2	526a	4.7	358b	3.9	482a	3.7	595c	11.3
borneol	1153	99ab	17.1	107a	2.1	69c	13.2	81bc	8.2	100ab	12.8
4-terpineol	1155	58a	7.9	73b	3.0	450c	7.1	58a	6.0	59a	5.0
dihydrocarvone	1177	736a	9.0	672ab	9.9	556b	4.1	622b	11.3	565b	8.5
<i>cis</i> -dihydrocarveol	1182	1733a	14.5	1434b	11.2	1111c	5.2	1109c	8.9	1561ab	9.2
<i>cis</i> -carveol	1205	115a	9.1	60bc	10.7	52b	9.3	70c	10.5	58bc	7.7
carvone	1228	14399a	7.1	14702a	5.2	11680b	6.9	14229a	5.5	15324a	9.4
endobornyl acetate	1275	85a	13.1	98b	3.2	74ac	13.0	65c	6.0	74ac	10.0
dihydroedulan	1290	42ab	17.5	50a	3.8	49a	12.5	40ab	11.2	38b	11.3
<i>trans</i> -dihydrocarvyl acetate	1314	430a	9.8	443a	9.3	381a	9.9	286b	11.6	525c	6.5
<i>cis</i> - <i>p</i> -mentha-6,8-dien-2-ol acetate	1320	87a	13.9	83a	10.9	72a	10.8	51b	10.1	77a	9.2
<i>trans</i> -carvyl acetate	1348	80a	14.0	103b	6.0	85a	9.3	63c	8.2	79a	7.6
<i>cis</i> -jasnone	1373	100a	9.9	105a	6.4	83b	4.9	65c	7.8	87b	6.4
β -bourbonene	1392	303a	14.7	295ab	6.0	278ab	14.0	271ab	14.3	225b	10.3
β -elemene	1394	79ab	15.1	85a	12.3	64b	12.1	87a	11.8	64b	8.7
β -caryophyllene	1428	534a	10.9	481ab	5.8	432b	11.4	452b	3.8	406b	8.7
β -selinene	1461	31a	10.7	28ab	9.4	24bc	10.4	19c	7.2	21c	12.5
sesquiterpene	1469	71a	15.5	63ab	6.5	54bc	9.7	50c	8.0	49c	12.2
<i>epi</i> -bicyclosesquiphellandrene	1487	425a	14.3	377ab	5.7	346bc	8.8	336bc	8.5	296c	5.5

^a Different letters (a, b, c, d) in the same row indicate statistical differences at the $\alpha = 0.05$ level according to the Student–Newman–Keuls test.

**Figure 1.** Scanning electron micrographs of the upper epidermis of fresh and dried spearmint leaves ($\times 500$).

sesquiterpenes. Sometimes substances of relatively low volatility, like sesquiterpenes, are released more readily than more volatile compounds. Certain researchers have suggested that there may be membranes selectively more permeable to certain volatiles or separate compartments for the synthesis of emitted volatiles and stored substances (25, 26).

Table 2. Volatiles Most Closely Correlated with Principal Components 1 and 2

PC	volatile compound	loading ^a	% explained variance	% cumulative variance
1	1,8-cineole + limonene	0.941	33.738	33.738
	β -myrcene	0.929		
	β -pinene	0.918		
	α -thujene	0.901		
	camphene	0.899		
	α -pinene	0.872		
	sabinene	0.820		
2	<i>epi</i> -bicyclosesquiphellandrene	0.949	33.671	67.410
	β -selinene	0.922		
	β -caryophyllene	0.872		
	sesquiterpene	0.866		
	<i>cis</i> -jasnone	0.823		

^a Only those volatiles with absolute correlation coefficient values >0.82 have been included.

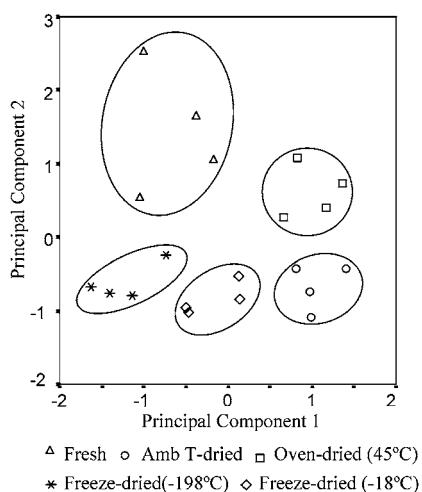
Figure 1e reveals expansion of the structure of the epidermis in these samples, probably as a result of differences between the internal sample steam pressure and the vacuum pressure in the freeze-dryer. Thus, the cell structure in these samples (frozen at -198 °C) is similar to that in the fresh samples. A similar expansion effect has been observed by other researchers during the microwave-drying of basil and carrots (14, 27). This effect may cause volatiles to be released into the air, emission into the atmosphere being one of the main ways of loss of volatile compounds in plants (28).

Table 2 presents the PCA results. The first two principal components explained 67% of the variance. **Figure 2** depicts sample distribution on the coordinate grid defined by these two components. Principal component 1 (33.7%) separated the oven-dried and ambient temperature-dried samples from the fresh

Table 3. Mean Scores for the Descriptors Most Closely Correlated with the First Two GPA Dimensions by Sample^a

	spearmint	herbaceous ($n = 10$) ^b	minty ($n = 10$)	fresh ($n = 8$)	earthy ($n = 9$)	sweet ($n = 8$)	floral ($n = 6$)	spicy ($n = 5$)
fresh		6.93a (1.72) ^c	2.21a (1.30)	5.93a (1.20)	0.00a (0.00)	1.00a (0.44)	3.70a (1.39)	1.30a (0.85)
oven-dried at 45 °C		0.11b (0.18)	7.84b (0.63)	0.38b (0.09)	3.89b (2.11)	4.90b (0.67)	0.48b (0.22)	0.33a (0.35)
ambient temp-dried		0.27b (0.49)	7.02bc (1.83)	2.80c (1.58)	4.85b (1.87)	5.11b (0.25)	0.75b (0.17)	0.53a (0.46)
freeze-dried (−18 °C)		0.81b (1.20)	5.94c (1.18)	1.93bc (0.53)	4.41b (1.57)	4.66b (0.45)	0.00b (0.00)	3.87b (0.15)
freeze-dried (−198 °C)		0.34b (0.63)	5.72c (1.96)	1.59bc (0.46)	5.88b (1.57)	4.98b (0.58)	0.45b (0.13)	3.07b (0.81)

^a Different letters (a, b, c) in the same column indicate statistical differences at the $\alpha = 0.05$ level according to the Student–Newman–Keuls test. ^b n = number of assessors that use those attributes. ^c Standard deviations in parentheses.

**Figure 2.** PCA of fresh and dried spearmint samples.

samples and the freeze-dried samples frozen at -198 °C. This principal component was correlated with the monoterpenes, the concentrations of which increased both in the samples oven-dried at 45 °C and in the ambient temperature-dried samples.

Principal component 2 explained 33% of the sample variance and separated the fresh samples and the samples oven-dried at 45 °C from the rest. It was most closely correlated with five sesquiterpenes, the concentrations of which decreased in the freeze-dried samples and in the samples dried at ambient temperature.

All of the samples produced by the different drying methods had differentiating features. However, ambient temperature and oven-dried samples presented similar characteristics. On the other hand, freeze-dried samples were also similar, although freeze-drying with freezing at -18 °C caused irreparable cell damage that had a major effect on the results.

Table 3 summarizes the results of the sensory analysis of the samples and gives the mean scores for the descriptors most commonly used by each assessor, which were most closely correlated with the first two dimensions in the GPA.

The assessors were able to differentiate the fresh samples from the dried samples, irrespective of the drying method employed, because of the stronger herbaceous, fresh, and floral aromas in the fresh samples. Mint odor was strongest in the samples oven-dried at 45 °C and in the samples dried at ambient temperature, and this may have been related to the higher monoterpene and carvone contents in these samples. Two attributes, namely, earthy odor and sweet odor, were described only for the dried samples, not for the fresh samples. These two attributes and the attribute hay odor have frequently been associated with dried spices. In dried parsley, the hay odor has been associated with the component 3-methyl-2,4-nonanedione, which forms in the presence of air (29).

No sensory differences were reported between freeze-dried and air-dried samples of dill (30), but in the spearmint some of

the assessors were able to distinguish the freeze-dried samples from the air-dried samples on the basis of the stronger spicy aroma of the former.

In conclusion, air-drying of spearmint both at ambient temperature and at 45 °C affected the cell structure of the samples, releasing higher quantities of monoterpenes and carvone, thus giving rise to a stronger mentholated aroma in these samples. Freeze-drying caused expansion of the surface layer of cells, causing volatiles, chiefly sesquiterpenes, to be lost. The results obtained by freeze-drying do not justify the high cost of this treatment, not even when the freezing is carried out at -198 °C to prevent cell damage of the spearmint leaves. Because drying at ambient temperature takes longer and the drying conditions are difficult to control, oven-drying at 45 °C would seem to be more advisable, in that this drying treatment is fast, simple, and inexpensive but affords control over the drying conditions, thereby helping to conserve the characteristic aroma of this spice.

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