Drying and Shade Effects on Spearmint Oil Yields and Composition

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Abstract. ‘Native’ spearmint (Mentha spicata L.) is one of the two most widely grown spearmints in the United States and in other countries. Recent studies demonstrated the feasibility of growing ‘Native’ spearmint as a cash crop for north–central Wyoming. Transportation and energy costs of commercial mint production can be reduced by drying the spearmint in windrows in the field for a few days after harvest and before oil extraction. This method of drying mint has been a common practice in the traditional mint production regions of the world. However, there is a knowledge gap regarding the effect of this drying method on the yield and composition of ‘Native’ spearmint oil. Field and laboratory experiments were conducted in Wyoming to evaluate the effects of drying duration in days after harvest (DAH: 0, 1, 2, 3, 4, 7, or 11) and drying conditions (shade and sun) on the yield of essential oil (EO) and on the concentrations of different oil constituents (beta-pinene, myrcene, limonene, eucalyptol, cis-sabinene hydrate, 4-terpineol, cis-dihydro carvone, cis-carveol, carvone, iso-dihydro carvone acetate, beta-bourbonene, beta-caryophyllene, alpha-humulene/trans-beta-farnesene, and germacrene D). Neither drying duration nor drying condition had a significant effect on oil yield. The average yield of essential oil was 0.25 g of oil per 100 g of fresh weight. Drying duration and drying conditions had a significant effect on the composition of EOs. The concentrations of myrcene and germacrene-D were higher in the EOs from plants dried under shade (3.2% and 2.4%, respectively) than the EOs from plants dried under direct sun (3% and 2.2%, respectively). The concentration of beta-pinene was higher in plants dried under direct sun than under shade (0.92% vs. 0.88%). Carvone ranged from 51% to 53% in the oil and was higher in EOs from plants dried for 1 and 2 DAH and lower in EOs from plants dried for 7 days. Drying of ‘Native’ spearmint under direct sun in Wyoming for up to 11 DAH can be used as an effective tool to reduce transportation and energy costs without affecting oil yields.

The United States is a major producer of EO from peppermint (Mentha ×piperita L.) and spearmints such as ‘Native’ spearmint (Mentha spicata L.) and ‘Scotch’ spearmint (Mentha ×gracilis Sole.; syn. M. cardiaca L.) [Lawrence, 2006; Mint Industry Research Council, 2012; National Agricultural Statistics Service (NASS), 2009]. These mint species are also grown in other countries in Europe, Australia, and New Zealand (Bienvenu et al., 1999; Lawrence, 2006). The production of ‘Native’ spearmint in the United States is concentrated in the midwest and northwestern states (NASS, 2009) and there has been an interest in expanding the production of ‘Native’ spearmint to other regions. Recent studies in Mississippi and Wyoming demonstrated that ‘Native’ spearmint can be grown in a wide range of environments outside the traditional spearmint production areas and produce desirable yields and oil composition (Zheljazkov et al., 2010a, 2010b, 2012, 2013). Spearmint oil content in fresh herbage is ≥0.15% to 0.3% and depends on many factors. For example, in studies conducted in Wyoming, the oil content of fresh spearmint varied from 0.14% to 0.22% with carvone concentration in the oil ranging between 42% and 75% depending on the timing of harvesting after the first fall frosts (Zheljazkov et al., 2013). In studies conducted in Mississippi with the same cultivar of spearmint, the carvone concentration was 36% to 63% in the oil of freshly distilled biomass and 45% to 62% in the oil of dried biomass at different harvesting times (Zheljazkov et al., 2010b). In another study conducted in Mississippi with the same cultivar of spearmint, the oil content in the fresh biomass was 0.18% to 0.19%, whereas the concentration of carvone in the oil varied between 68 and 74% (Zheljazkov et al., 2010a).

In the spearmint and peppermint production industry, transportation and energy are significant components of the total cost. To decrease both transportation and energy costs associated with production and distillation (the extraction of the EO), commercial mint producers have been drying the spearmint in windrows in the field for 2 to 7 DAH and before oil extraction. Drying reduces moisture content of harvested biomass, decreasing the biomass weight three to four times relative to the weight of fresh herbage at harvest. Hence, both transportation and the energy costs associated with the distillation are reduced. As a result of the reduced moisture content, more dried biomass can be fitted into the distillation units relative to the fresh herbage biomass, which increases efficiency of distillation. However, there is a knowledge gap regarding the effect of drying on ‘Native’ spearmint oil yields and composition. There have been some previous studies on the effect of different drying methods (oven-drying, freeze-drying, and air-drying) on spearmint flavor and oil composition (Diaz-Maroto et al., 2003). However, this is not how the mint industry is drying spearmints or peppermints before oil extraction. Zheljazkov et al. (2010b) reported oil compositional differences between oils extracted from dried vs. fresh biomass of the same cultivar of spearmint harvested at different times (and developmental stages). However, neither of these studies provided insight on how drying duration and drying condition (shade and sun) affect the essential oil yield and composition of ‘Native’ spearmint. Therefore, the objective of this study was to determine the effects of drying duration in DAH (0, 1, 2, 3, 4, 7, and 11) and drying condition (shade and sun) on the EO yield and on the concentrations of 47 major and minor constituents in the oil.

Materials and Methods

Field and laboratory experiments. The field and laboratory experiments were conducted in 2012 at the Sheridan Research and Extension Center Experimental Fields at latitude 44°45′686″ N and longitude –106°55′479″ W, elevation 1171 m above sea level. The plantation with ‘Native’ spearmint was established in 2011 using certified virus-free ‘Native’ spearmint transplants from Summit Plant Laboratories, Inc. (Fort Collins, CO). The tissue culture propagation of the plant material ensures that all individual plants have the same genotype and are free of viral diseases.
Land preparation, planting, fertilization, and irrigation. Land preparation and transplanting were carried out as described earlier (Zheljazkov et al., 2012, 2013). Raised beds were prepared with a press-pan-type bed-shaper machine. Spearmint transplants (10 to 12 cm tall) were transplanted at in-row and between-row spacing of 30 cm in an offset pattern. Transplanting was done in the spring of 2011; this study was conducted in 2012 on a 2-year well-established spearmint plantation. Twinbar (Tercabil 80% WP, DuPont, Wilmington, DE) at the rate of 2 kg/ha⁻¹ was applied for weed control before transplanting and incorporated through irrigation. This herbicide is traditionally used in spearmint and peppermint production fields across the United States and worldwide (Lawrence, 2006; Mint Industry Research Council, 1967).

Additionally, spearmint plots were hand-weeded twice to remove weeds unaffected by the applied herbicide. Irrigation was provided through a low-pressure drip-tape system calculated to deliver 2.5 cm water/week (0.2 mm, emitters spaced at 30 cm, 1703 cm²/min/30.5 m). Nitrogen (ammonium nitrate) was surface-applied by side-dressing when plants were 10 to 12 cm tall at 180 kg nitrogen/ha.

Harvesting and drying. All samples were obtained on 16 July 2012 by harvesting the flowering spearmint plants at 4 to 5 cm above the soil surface using a hedge trimmer. Forty-two 1-kg fresh spearmint biomass samples were generated (7 DAH × two drying conditions × three replications). The fresh spearmint samples were dried either in direct sun or in a shady place (in a well-ventilated barn) until the oil was extracted.

The spearmint samples were distilled at the following DAH: 0 (distillated 3 h after harvest on the same day), 1, 2, 3, 4, 7, and 11 DAH. During this 11-d period, the night air temperatures fluctuated between 11 and 19 °C. The daytime temperatures during this period were between 29 and 37 °C. Also during this 11-d period, there were 24 mm of rain on 17 July (1 DAH) and 1 mm on 18 July (2 DAH). Samples that were drying under rain on 17 July (1 DAH) and 1 mm on 18 July (2 DAH) were transplanted at in-row and between-row spacing of 30 cm in an offset pattern. Transplanting was done in the spring of 2011; this study was conducted in 2012 on a 2-year well-established spearmint plantation. Twinbar (Tercabil 80% WP, DuPont, Wilmington, DE) at the rate of 2 kg/ha⁻¹ was applied for weed control before transplanting and incorporated through irrigation. This herbicide is traditionally used in spearmint and peppermint production fields across the United States and worldwide (Lawrence, 2006; Mint Industry Research Council, 1967).

**Gas chromatography of spearmint essential oils.** The spearmint EO samples were analyzed for chemical profile on a Hewlett Packard (Hewlett-Packard, Palo Alto, CA) gas chromatograph (GC) Model 6890. The carrier gas was helium, 40 cm/sec., 11.7 psi (60 °C), 2.5 mL/min constant flow rate; the injection was split 60:1 with 0.5 μL, the injector temperature was 220 °C; the oven temperature program was 60 °C for 1 min and 10 °C/min to 250 °C. The GC column was HP-INNOWAX (Agilent Technologies, Santa Clara, CA) (the column was crosslinked polyethylene glycol; 30 m × 0.32 mm × 0.5 μm), and the flame ionization detector (FID) temperature was 275 °C.

Identification of different oil constituents. The identification of individual peaks was done using internal standards (for all the major constituents) by retention time and also using mass spectroscopy (MS). The constituents identified on MS were beta-pinene, myrcene, limonene, eucalyptol, cis-sabinene hydrate, 4-terpinol, cis-dihydro carvone, cis-carveol, carvone, iso-dihydro carvone aceta, beta-bourbonene, beta-carphyllyene, and germacrene D. Of the 47 oil constituents identified (Table 1), we selected the 14 with the highest concentration (beta-pinene, myrcene, limonene, eucalyptol, cis-sabinene hydrate, 4-terpinol, cis-dihydro carvone, cis-carveol, carvone, iso-dihydro carvone aceta, beta-bourbonene, beta-carphyllyene, alpha-humulene/transbeta-farnesene, and germacrene D) for statistical analyses and report.

**Statistical analysis.** The effects of drying duration (0, 1, 2, 3, 4, 7, and 11 DAH) on drying condition (shade and sun) on EO content and the concentrations of beta-pinene, myrcene, limonene, eucalyptol, cis-sabinene hydrate, 4-terpinol, cis-dihydro carvone, cis-carveol, carvone, iso-dihydro carvone acetate, beta-bourbonene, beta-carphyllyene, alpha-humulene/transbeta-farnesene, and germacrene D were determined using a seven (2 × 7) factorial design (Montgomery, 2013) with three replications. The analysis of variance of all these response measurements was completed using the GLM Procedure of SAS (SAS Institute Inc., 2010). When the interaction effect is significant (P < 0.05), the significance of the main effects was ignored and multiple means comparison was completed by comparing the least squares means of the corresponding 14 (seven × two) treatment combinations at the 1% level of significance to protect the Type I experiment-wise error rate from overinflation resulting from the relatively large number of means being compared. However, when the interaction effect is not significant, multiple means comparison of the significant main effect was completed by comparing the least squares means of the 7 DAH and/or the two drying conditions at the 5% level of significance. The effects that required these multiple means comparison are shown as * or ** in Table 2. For each response variable, the validity of normal distribution and constant variance model assumptions was verified by examining the residuals as described in Montgomery (2013).

Correlation analysis was also completed to determine the type (positive or negative) and the strength of the relationships among EO content and the concentrations of the constituents.

Table 1. Concentration ranges of the 47 oil constituents in percent of total oil.

| Constituent                        | Range (%) | Constituent                  | Range (%) |
|-----------------------------------|-----------|------------------------------|-----------|
| Beta-pinene                       | 0.30–1.03 | Beta-copaene                 | 0.21–0.33 |
| Myrcene                           | 2.58–3.91 | Gamma-terpinene              | 0.21–0.73 |
| Limonene                          | 10.25–14.86 | 3-Octanol acetate           | 0.27–0.46 |
| Eucalyptol                        | 1.14–1.87 | Dihydro carvone (and-or)     | 0.28–0.70 |
| cis-sabinene hydrate              | 1.11–3.35 | Trans-carveol                | 0.28–0.71 |
| 4-terpinol                        | 0.29–1.23 | Viridifloral (and-or) Globulol | 0.41–0.89 |
| cis-dihydro carvone               | 1.48–3.41 | Trans-caryl acetate          | 0.44–0.87 |
| cis-carveol                       | 4.25–7.14 | Cis-jasmone                  | 0.56–0.78 |
| Carvone                           | 49.78–54.85 | Sabine               | 0.62–0.80 |
| iso-dihydro carvone acetate       | 0.46–1.32 | 3-octanol                   | 0.62–0.94 |
| Beta-bourbonene                   | 1.86–3.10 | Alpha-pinene                | 0.88–0.67 |
| Beta-caryophyllene                | 1.67–2.42 | 2-ethyl furan; Ethyl-2-methyl butyrate | ND–0.03 |
| Alpha-humulene/transbeta-farnesene| 1.07–1.42 | Pinocarvone                 | ND–0.07 |
| Germacrene D                      | 1.91–2.83 | Para-cymene; Transpinocarvone | ND–0.08 |
| Terpinolene                       | 0.10–0.24 | 2-methyl butyl-2-methyl butyrate | ND–0.09 |
| Trans-ocimene                     | 0.10–0.30 | Eugenol                      | ND–0.09 |
| Alpha-terpinol                    | 0.11–0.44 | Alpha-thujene; 1-octanal/Delta-2-carene; Translimonene oxide; Transverbenol; alpha-copaene; germacrene A | ND–0.10 |
| Alpha-terpinol                    | 0.14–0.20 | cis-carveone oxide           | ND–0.12 |
| Piperitone                         | 0.14–0.23 | cis-3-hexenol                | ND–0.13 |
| Gamma-muurolene                   | 0.15–0.22 | Trans-2-hexanal              | ND–0.14 |
| Delta-terpinol                    | 0.16–0.19 | Methyl-2-methyl butyrate      | ND–0.15 |
| Trans-sabinene hydrate            | 0.17–0.24 | Trans-carve oxide            | ND–0.20 |
| cis-ocimene                       | 0.15–0.34 | Caryophyllene oxide          | ND–0.28 |
| Trans-jasmonene/ beta-clemon      | 0.21–0.29 | 

*ND = not detected.*
Results and Discussion

The main and interaction effects of drying duration and drying condition on essential oil yield and composition. Drying duration had a significant effect on the concentrations of beta-pinene, 4-terpinol, carvone, isodihydro carveol acetate, alpha-humulene/transbeta-farnesene, and germacrene D regardless of drying condition (no significant interaction) (Table 2). Drying conditions (sun or shade) had a significant main effect on the concentrations of beta-pinene, myrcene, and germacrene D, whereas the interaction of drying duration and drying condition was significant on the concentrations of limonene, eucalyptol, cis-sabinene hydrate, cis-dihydro carveol, beta-bourbonene, and beta-caryophyllene (Table 2). Neither drying duration nor drying condition affected essential oil content (0.25±4% average in fresh herbage) or the concentration of cis-carveol in the oil (5.96±4% average).

Effect of drying duration and drying conditions on the concentration of oil constituents. Drying of the spearmint biomass under direct sun increased the concentration of beta-pinene but reduced the concentrations of myrcene and germacrene D in the oil relative to drying under shade (Table 3). The concentration of beta-pinene in the oil was low at 0 DAH (distilled the same day after 3 h) and higher at 3, 7, and 11 DAH (Table 3). The concentration of carvone, the main oil constituent, was higher in the oil at 1 or 2 DAH than at 7 DAH and not significantly different from that at 0, 3, 4, and 11 DAH. The concentration of isodihydro carveol acetate was high in the oil at 0 DAH but only higher than DAH 1 and 2 and similar to the other DAHs. The combined concentrations of alpha-humulene/transbeta-farnesene were low in the oil at 0 DAH and increased in the oils from 2 to 11 DAH. The concentration of germacrene D was lower in the oil at 0 DAH than at 2, 4, and 11 DAH (Table 3).

Interaction effect of drying duration and drying conditions on the concentration of oil constituents. The concentration of limonene in the oil was highest at 0 DAH and lowest at 11 DAH under sun (Table 4). Conversely, the concentration of eucalyptol was low in the oil at 0 DAH and higher in the oil at 11 DAH under shade (Table 4). The concentration of cis-sabinene hydrate was low in the oils at 0 DAH and high at 4 to 11 DAH under shade and at 11 DAH under sun (Table 4). The concentration of cis-sabinene hydrate was low in the oils at 0 DAH and increased with increasing drying period under sun but not under shade to reach a maximum concentration in the oil at 11 DAH under sun (Table 4). The concentration of beta-caryophyllene (1.71% to 2.26% range) was also low in the oils at 0 DAH and generally increased with increasing drying period to reach a maximum concentration in the oil at 11 DAH under sun (Table 4).

The main and interaction effects of drying duration (DAH) and drying condition (Drying) on essential oil (EO) content and the concentrations of beta-pinene, myrcene, eucalyptol, cis-sabinene hydrate, 4-terpinol, cis-dihydro carvone, cis-carveol, carvone, isodihydro carveol acetate, beta-bourbonene, beta-caryophyllene, alpha-humulene/transbeta-farnesene, and germacrene D.

### Table 2

| EO content and constituents | DAH | Drying | DAH*drying |
|-----------------------------|-----|--------|-------------|
| **EO content**              |     |        |             |
| *                            | NS  | NS     | NS          |
| **Beta-pinene**             |     |        | NS          |
| NS                          | NS  | NS     | NS          |
| Myrcene                     | NS  | NS     | NS          |
| NS                          | NS  | NS     | NS          |
| Limonene                    | NS  | NS     | NS          |
| NS                          | NS  | NS     | NS          |
| Eucalyptol                  | NS  | NS     | NS          |
| NS                          | NS  | NS     | NS          |
| Cis-sabinene hydrate        | NS  | **     | NS          |
| NS                          | NS  | NS     | NS          |
| 4-terpinol                  | NS  | **     | NS          |
| NS                          | NS  | NS     | NS          |
| Cis-dihydro carveol         | NS  |        | *           |
| NS                          | NS  | NS     | NS          |
| Cis-carveol                 | NS  |        | *           |
| NS                          | NS  | NS     | NS          |
| Carvone                     | NS  |        | NS          |
| NS                          | NS  | NS     | NS          |
| Iso-dihydro carvone acetate | **  | NS     | NS          |
| NS                          | NS  | NS     | NS          |
| Beta-bourbonene             | NS  |        | NS          |
| NS                          | NS  | NS     | NS          |
| Beta-caryophyllene          | NS  |        | **          |
| NS                          | NS  | NS     | NS          |
| Alpha-humulene/transbeta-farnesene | **  | NS  | NS          |
| NS                          | NS  | NS     | NS          |
| Germacrene D                | *   |        | **          |
| **                          | NS  | NS     | NS          |

*ns = Either the effect is nonsignificant (P > 0.05) or its significance is ignored because the interaction effect is significant.
** Significant at α = 5%, 1%, respectively, and require multiple means comparison.
ANOVA = analysis of variance.

### Table 3

| Factor                | Beta-pinene | Myrcene | Carvone | Iso-dihydro carveol acetate | Alpha-humulene/transbeta-farnesene | Germacrene D |
|-----------------------|-------------|---------|---------|----------------------------|----------------------------------|--------------|
| Drying Shade          |             |         |         |                            |                                  |              |
| 0                     | 0.881 b     | 3.20 a  | 52.4 a  | 0.877 a                    | 1.24 a                           | 2.43 a        |
| Sun                   | 0.921 a     | 3.01 b  | 51.9 a  | 0.850 a                    | 1.22 a                           | 2.22 b        |
| DAH                   |             |         |         |                            |                                  |              |
| 0                     | 0.848 c     | 52.6 a  | 1.075 b | 1.10 b                     | 2.15 b                           |              |
| 1                     | 0.897 bc    | 52.8 a  | 0.813 b | 1.18 ab                    | 2.26 ab                          |              |
| 2                     | 0.883 bc    | 52.7 a  | 0.758 b | 1.25 a                     | 2.41 a                           |              |
| 3                     | 0.913 ab    | 52.1 a  | 0.867 b | 1.25 a                     | 2.32 ab                          |              |
| 4                     | 0.887 bc    | 51.9 a  | 0.867 ab| 1.29 a                     | 2.41 a                           |              |
| 5                     | 0.960 a     | 51.0 b  | 0.842 ab| 1.26 a                     | 2.32 ab                          |              |
| 6                     | 0.918 ab    | 51.8 ab | 0.823 ab| 1.29 a                     | 2.42 a                           |              |

*Factor levels whose means do not share the same letter are significantly different.

### Table 4

| DAH | Drying | Limonene | Eucalyptol | Cis-sabinene hydrate | Cis-dihydro carveol | Beta-bourbonene | Beta-caryophyllene |
|-----|--------|----------|------------|---------------------|--------------------|----------------|-------------------|
| 0   | Shade  | 13.5 a   | 1.33 dc    | 1.42 f              | 3.05 ab            | 1.90 g         | 1.71 f            |
|     | 1 Shade | 12.8 abc | 1.37 cde   | 1.71 ef             | 2.53 abc           | 2.12 efg        | 1.94 cde          |
|     | 2 Shade | 12.4 abc | 1.42 bcd   | 1.97 cdef           | 2.45 ab            | 2.14 efg        | 1.96 bcd          |
|     | 3 Shade | 12.6 abc | 1.41 cde   | 2.13 bcde           | 2.60 abc           | 2.20 efg        | 2.01 bcd          |
|     | 4 Shade | 11.4 bc  | 1.54 bc    | 2.96 a              | 3.23 a             | 2.23 def        | 1.98 bcd          |
|     | 5 Shade | 12.5 abc | 1.50 bcd   | 2.72 ab             | 2.67 abc           | 2.14 efg        | 1.94 cde          |
|     | 6 Shade | 13.3 ab  | 1.43 bcd   | 2.50 abc            | 1.87 c             | 2.10 efg        | 1.94 cde          |
|     | 7 Sun   | 13.8 a   | 1.21 c     | 1.61 ef             | 2.78 abc           | 1.96 fg         | 1.75 ef           |
|     | 8 Sun   | 13.0 abc | 1.46 bcd   | 1.79 def            | 2.66 abc           | 2.11 efg        | 1.82 def          |
|     | 9 Sun   | 13.0 abc | 1.38 cde   | 1.89 def            | 2.46 abc           | 2.38 cde        | 2.02 bcd          |
|     | 10 Sun  | 13.4 ab  | 1.39 cde   | 2.34 bcd            | 2.67 abc           | 2.49 cd         | 2.03 bcd          |
|     | 11 Sun  | 13.2 ab  | 1.42 bcd   | 2.54 abc            | 2.13 bc            | 2.58 bc         | 2.09 abc          |
|     | 12 Sun  | 12.9 abc | 1.61 b     | 2.34 bcd            | 2.49 abc           | 2.78 ab         | 2.17 ab           |
|     | 13 Sun  | 11.0 c   | 1.82 a     | 3.03 a              | 2.68 abc           | 2.87 a          | 2.26 a            |

*Means that do not share the same letter are significantly different.
DAH = days after harvest.
correlated to eucalyptol, cis-sabinene hydrate, cis-dihydro carvone, beta-caryophyllene, alpha-humulene/trans beta-farnesene, and negatively to eucalyptol, cis-sabinene hydrate, cis-dihydro carvone, beta-caryophyllene, alpha-humulene/trans beta-farnesene, and germacrene. Eucalyptol was positively correlated to cis-sabinene hydrate, beta-bourbonene, beta-caryophyllene, and alpha-humulene/trans beta-farnesene and negatively correlated to 4-terpineol and carvone. Cis-sabinene hydrate was positively correlated to beta-bourbonene, beta-caryophyllene, alpha-humulene/trans beta-farnesene, and germacrene D and negatively correlated to 4-terpineol and carvone. Terpineol was negatively correlated to cis-carveol, beta-bourbonene, beta-caryophyllene, alpha-humulene/trans beta-farnesene, and germacrene D. Cis-dehydro carvone was positively correlated to iso-dihydro carveol acetate and negatively correlated to cis-carveol. Cis-carveol was positively correlated to carvone and negatively correlated to iso-dihydro carveol acetate. Carvone was negatively correlated to iso-dihydro carveol acetate, beta-bourbonene, beta-caryophyllene, and alpha-humulene/trans beta-farnesene. Beta-bourbonene was positively correlated to beta-caryophyllene and alpha-humulene/trans beta-farnesene. Beta-caryophyllene was positively correlated to alpha-humulene/trans beta-farnesene and germacrene D. The concentration of alpha-humulene/trans beta-farnesene was positively correlated to germacrene D (Table 5).

Concluding Discussion

In this study the concentration of carvone varied from 51% to 52.8%, which was within the range reported previously for ‘Native’ spearmint grown in Wyoming (Zheljazkov et al., 2012, 2013) and other regions in the United States (Murray et al., 1972; Zheljazkov et al., 2010a, 2010b). For example, carvone concentration in the oil of the same cultivar of spearmint reported by Zheljazkov et al. (2013) varied from 51.4% to 55.9%, whereas carvone concentration in Zheljazkov et al. (2012) varied from 42.9% to 74.6%. Carvone concentration in this study was also comparable to literature reports from other countries (Bienvenu et al., 1999; Díaz-Maroto et al., 2003; Kizil and Toncer, 2006; Sokovic et al., 2009; Topalov, 1989). Overall, the concentration of carvone in spearmint oils is expected to be above 50% of the oil (de Carvalho and Da Fonseca, 2006).

Another example was beta-caryophyllene concentration: the concentration of beta-caryophyllene in the study of Zheljazkov et al. (2013) varied from 5.4% to 55.9%, whereas carvone concentration in Zheljazkov et al. (2012) varied from 42.9% to 74.6%. Carvone concentration in this study was also comparable to literature reports from other countries (Bienvenu et al., 1999; Díaz-Maroto et al., 2003; Kizil and Toncer, 2006; Sokovic et al., 2009; Topalov, 1989). Overall, the concentration of carvone in spearmint oils is expected to be above 50% of the oil (de Carvalho and Da Fonseca, 2006).

Our results demonstrated that although the actual EO content (relative to the initial fresh biomass sample) may not change as a function of drying duration (0 to 11 DAH) or drying conditions (sun or shade), drying would increase the amount of oil obtained from the dried sample as a result of decreased moisture...
in the sample. Therefore, drying may reduce transportation and energy costs associated with distillation of the dried biomass vs. fresh biomass without compromising EO content. Although the duration of drying and the condition of drying significantly affected the concentrations of a number of oil constituents, these alterations were within the range of concentrations for individual oil constituents as reported in the literature for ‘Native’ spearmint oil. Therefore, these alterations may not affect the overall quality and the market price of the spearmint oil.

**Literature Cited**

Bienvenu, F., L. Peterson, and J. Edwards. 1999. Native and Scotch spearmint oil production in Tasmania and Victoria. A report for Rural Industries Research and Development Corporation. Publ. #99/147, Project #DAV-101A, p. 32. 11 June 2013. <https://rirdc.infoservices.com.au/downloads/99-147>.

de Carvalho, C.C.C.R. and M.M.R. Da Fonseca. 2006. Carvone: Why and how should one bother to produce this terpene. Food Chem. 95:413–422.

Díaz-Maroto, M.C., M.S. Pérez-Coello, M.A. González Viñas, and M.D. Cabezudo. 2003. Influence of drying on the flavor quality of spearmint (*Mentha spicata* L.). J. Agr. Food Chem. 51:1265–1269.

Gawde, A.J., C.L. Cantrell, and V.D. Zheljazkov. 2009. Dual extraction of essential oil and podophyllotoxin from *Juniperus virginiana*. Ind. Crops Prod. 30:276–280.

Kizil, S. and O. Toncer. 2006. Influence of different harvesting times on the yield and oil composition of spearmint (*Mentha spicata* var. *spicata*). J. Food Agr. Environ. 4:135–137.

Lawrence, B.M. 2006. Mint: The genus Mentha. CRC Press, Boca Raton, FL.

Mint Industry Research Council. 1967. Summary of research conducted with mint industry research council funds in 1967. July 2013. <http://usmintindustry.org/Portals/1/PDF/ID6411.pdf>.

Montgomery, D.C. 2013. Design and analysis of experiments. 8th Ed. Wiley, New York, NY.

Murray, M.J., W. Faas, and P. Marble. 1972. Effects of plant maturity on oil composition of several spearmint species grown in Indiana and Michigan. Crop Sci. 12:723–728.

National Agricultural Statistics Service. 2009. 11 June 2013. <http://www.nass.usda.gov/Statistics_by_State/Oregon/Publications/Field_Crop_Report/crop%20reports/01_13an.pdf>.

SAS Institute Inc. 2010. SAS/STAT® 9.3 user’s guide. SAS Institute Inc., Cary, NC.

Sokovic, M.D., J. Vukojevic, P.D. Marin, D.D. Brkic, V. Vajs, and L.J.L.D. van Griensven. 2009. Chemical composition of essential oils of *Thymus* and *Mentha* species and their antifungal activities. Molecules 14:238–249.

Topalov, V.D. 1989. *Mentha*, p. 372–381. In: Topalov, V.D., I.I. Dechev, and M.S. Pehivanov (eds.). Plant production. Zemizdat Press, Sofia, Bulgaria.

Zheljazkov, V.D., T. Astatkie, and E.A. Jeliázkova. 2013. Effect of foliar application of methyl jasmonate and extracts of juniper and sagebrush on essential oil yield and composition of ‘Native’ spearmint. HortScience 48:462–465.

Zheljazkov, V.D., C.L. Cantrell, T. Astatkie, and M.W. Ebelhar. 2010a. Productivity, oil content and composition of two spearmint species in Mississippi. Agron. J. 102:129–133.

Zheljazkov, V.D., C.L. Cantrell, T. Astatkie, and A. Hristov. 2010b. Yield, content, and composition of peppermint and spearmints as a function of harvesting time and drying. J. Agr. Food Chem. 58:11400–11407.

Zheljazkov, V.D., C.L. Cantrell, T. Astatkie, and E. Jeliázkova. 2012. Fall frosts effects on the essential oil of ‘Native’ spearmint in Wyoming. HortScience 47:1603–1606.