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Distillation Time Effect on Lavender Essential Oil Yield and Composition

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Abstract: Lavender (*Lavandula angustifolia* Mill.) is one of the most widely grown essential oil crops in the world. Commercial extraction of lavender oil is done using steam distillation. The objective of this study was to evaluate the effect of the length of the distillation time (DT) on lavender essential oil yield and composition when extracted from dried flowers. Therefore, the following distillation times (DT) were tested in this experiment: 1.5 min, 3 min, 3.75 min, 7.5 min, 15 min, 30 min, 60 min, 90 min, 120 min, 150 min, 180 min, and 240 min. The essential oil yield (range 0.5-6.8%) reached a maximum at 60 min DT. The concentrations of cineole (range 6.4-35%) and fenchol (range 1.7-2.9%) were highest at the 1.5 min DT and decreased with increasing length of the DT. The concentration of camphor (range 6.6-9.2%) reached a maximum at 7.5-15 min DT, while the concentration of linalool acetate (range 15-38%) reached a maximum at 30 min DT. Results suggest that lavender essential oil yield may not increase after 60 min DT. The change in essential oil yield, and the concentrations of cineole, fenchol and linalool acetate as DT changes were modeled very well by the asymptotic nonlinear regression model. DT may be used to modify the chemical profile of lavender oil and to obtain oils with differential chemical profiles from the same lavender flowers. DT must be taken into consideration when citing or comparing reports on lavender essential oil yield and composition.

Key words: steam distillation, essential oil yield, cineole, fenchol, camphor, linalool acetate

1 INTRODUCTION

Lavender (*Lavandula angustifolia* Mill.) is one of the most widely grown essential oil crops in the world, with major producing regions in Europe, the Middle East, Asia, and Northern Africa. Most of the lavender production is concentrated in Bulgaria and France, but other countries such as Morocco, former Yugoslavia, Hungary, Italy, Russia, Spain, Romania, Ukraine, and Turkey, also have significant production. In addition, lavender has been very popular with some small producers in the United States, either as part of agro-tourisms, entertainment farms, or aromatherapy businesses[1]. The extensive lavender oil production is due to its widespread uses by various industries, such as pharmaceutical, foods, beverages, liqueurs, perfumery and cosmetics, and ecofriendly pesticides[2, 3] Lavender oil has been shown to have antimicrobial properties[4-10]. Lavender essential oil has been tested as a natural product for suppression of methane production in cows; methane production was reduced by *Lavandula latifolia*, but not from *Lavandula angustifolia* essential oil[11]. Lavender oil also has been used as a natural plant growth regulator to suppress potato (*Solanum tuberosum* L.) sprouting[12]. Due to its highly aromatic and beautiful inflorescences with various colors, its wide ecological adaption and its drought resistance, lavender is also valued and is used in honey production and as an ornamental plant.

Lavender essential oil chemical composition (and subsequently its aroma and other properties) has been shown to be affected by genotype, environment, and processing[3, 13]. Lavender essential oil is traditionally extracted via steam distillation. We hypothesized that distillation time (DT) may also play a role in lavender essential oil composition. Also, there is no agreement in the literature as to the optimal DT for lavender essential oil extraction. Therefore, the objectives of this study were to evaluate the effect of DT on lavender essential oil yield and composition, and to develop a model that describes the relationship between DT and essential oil yield and composition.

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2 MATERIALS AND METHODS

2.1 Plant material and essential oil steam distillation

Bulk certified dried flowers of *Lavandula angustifolia* Mill. were purchased from Starwest Botanicals (Rancho Cordova, CA). The distillation study experiment was conducted at the University of Wyoming Sheridan Research and Extension Center in 2012, using 250 g of dried lavender flowers. Each DT was performed in 3 replicates. The essential oil was extracted via the steam distillation method (that is used for lavender oil extraction in commercial production) in 2-L steam distillation units, as described previously for peppermint and spearmint[14, 15]. The distillation times (DT) tested in this experiment were: 1.5 min, 3 min, 3.75 min, 7.5 min, 15 min, 30 min, 60 min, 90 min, 120 min, 150 min, 180 min, and 240 min. These DT were selected based on a preliminary study with lavender flowers, distillation experiments with other crops[16, 17], and literature reports. All DT were measured from the initiation of the actual distillation, as soon as the first drop of essential oil was visible, until the end of the DT, at which point the heating was turned off, the steam source removed, and the Florentine vessel (the apparatus part where the oil is separated from the water and accumulated) was also removed from the apparatus. Lavender oil was separated from the remaining water, measured on an analytical scale, and kept in a freezer at −5℃ until gas chromatography (GC) analyses. The oil yield was calculated as grams of oil per 100 g of dry lavender flowers.

2.2 Gas Chromatography Flame Ionization Detection (GC-FID) Essential Oil Quantitative Analysis

Oil samples were analyzed by GC-FID on a Varian CP-3800 GC equipped with a DB-5 fused silica capillary column (30 m × 0.25 mm, with a film thickness of 0.25 μm) operated using the following conditions: injector temperature, 240℃; column temperature, 60-240 at 3℃/min, then held at 240℃ for 5 min; carrier gas, He; injection volume, 1 μL (splitless); MS mass range, 40-650 *m/z*; filament delay, 3 min; target TIC, 20000; prescan ionization time, 100 μs; ion trap temperature, 150℃; manifold temperature, 60℃; and transfer line temperature, 170℃[18].

Compounds 1,8-cineole, fenchol (α or β undefined), (−)-camphor, and linalool acetate were identified in oil samples by Kovat analysis[19] and comparison of mass spectra with those reported in the NIST mass spectral database. The identity of 1,8-cineole and (−)-camphor were further verified using commercially available standards from Fluka (Buchs, Switzerland). Compounds were quantified by performing area percentage calculations based on the total combined FID area. For example, the area for each reported peak was divided by the total integrated area from the FID chromatogram from all reported peaks and multiplied by 100 to arrive at a percentage. The percentage is a peak area percentage relative to all other constituents integrated in the FID chromatogram.

2.3 Statistical analyses

The effect of distillation time on essential oil (EO) yield, and the concentration of cineole, fenchol, camphor, and linalool acetate was determined using a one-way analysis of variance. For each response, the validity of model assumptions was verified by examining the residuals as described previously[20]. Since the effect of distillation time was significant (p-value <0.05) on all responses, multiple means comparison was completed using Duncan's multiple range test at the 5% level of significance, and letter groupings were generated. The analysis was completed using the GLM Procedure of SAS[21].

The most appropriate model to describe the relationship between distillation time and EO yield, as well as the concentrations of cineole, fenchol and linalool acetate was the Asymptotic Regression model (Eq. 1), but with different parameters representing either increasing or decreasing patterns. However, there was no model that describes the relationship between camphor concentration and distillation time. Since the Asymptotic model is nonlinear that requires iterative estimation of the parameters, the NLIN Procedure of SAS (SAS Institute Inc. 2008) was used to estimate the parameters. The figures were generated using Minitab 16 software (Minitab, State College, PA, USA).

$$Y = \theta_1 - \theta_2(\exp(-\theta_3 x)) + \varepsilon \quad \text{(Eq. 1)}$$

Where Y is the dependent (response) variable, X is the independent (distillation time) variable, and the error term ε is assumed to have normal distribution with constant variance.

3 RESULTS

3.1 Essential oil yield as a function of DT

The major constituents of lavender oil were cineole, fenchol, camphor, and linalool acetate (Fig. 1). Distillation time had an effect on essential oil yield, and on the concentrations of fenchol, linalool acetate, and cineole (Fig. 2). The essential oil yield (concentration, range 0.5-6.8%) was low at 1.5-3.75 min DT, increased at 7.5 min DT, increased again at 15 and at 30 min and reached a maximum at 60 min DT (Table 1). Further increase in DT did not affect essential oil yield. The concentration of cineole (range 6.4-35%) was highest at the 1.5 min DT (35%) and decreased stepwise with the increase of DT to stabilized at minimum concentrations at 30-240 min DT (6.4-8.2%) (Table 1). The concentration of fenchol (range 1.7-2.9%) was also the highest at 1.5 min DT (2.9%), and decreased stepwise with the increase in DT of up to 30 min; further increase in DT did not change its concentration. The concentration of camphor (range 6.6-9.2%) was the lowest at

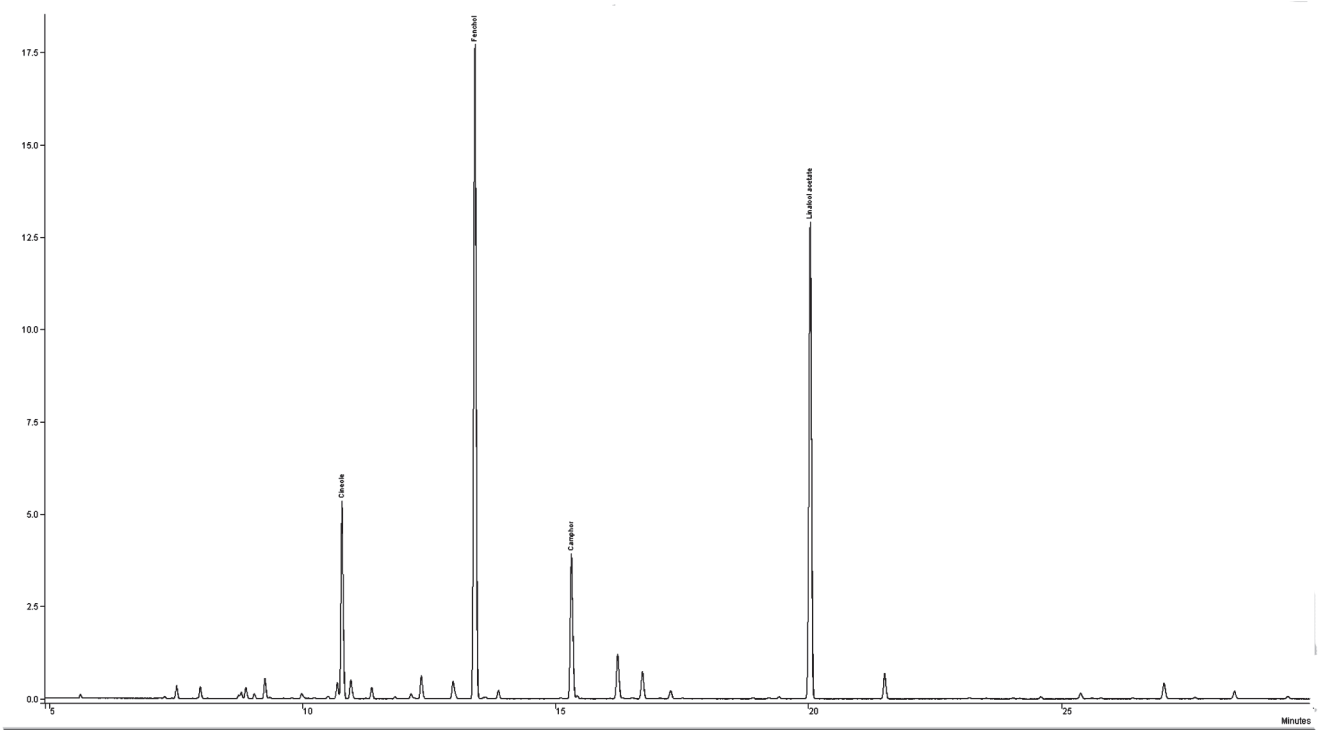

Fig. 1 Representative GC-FID chromatogram of *L. angustifolia* essential oil.

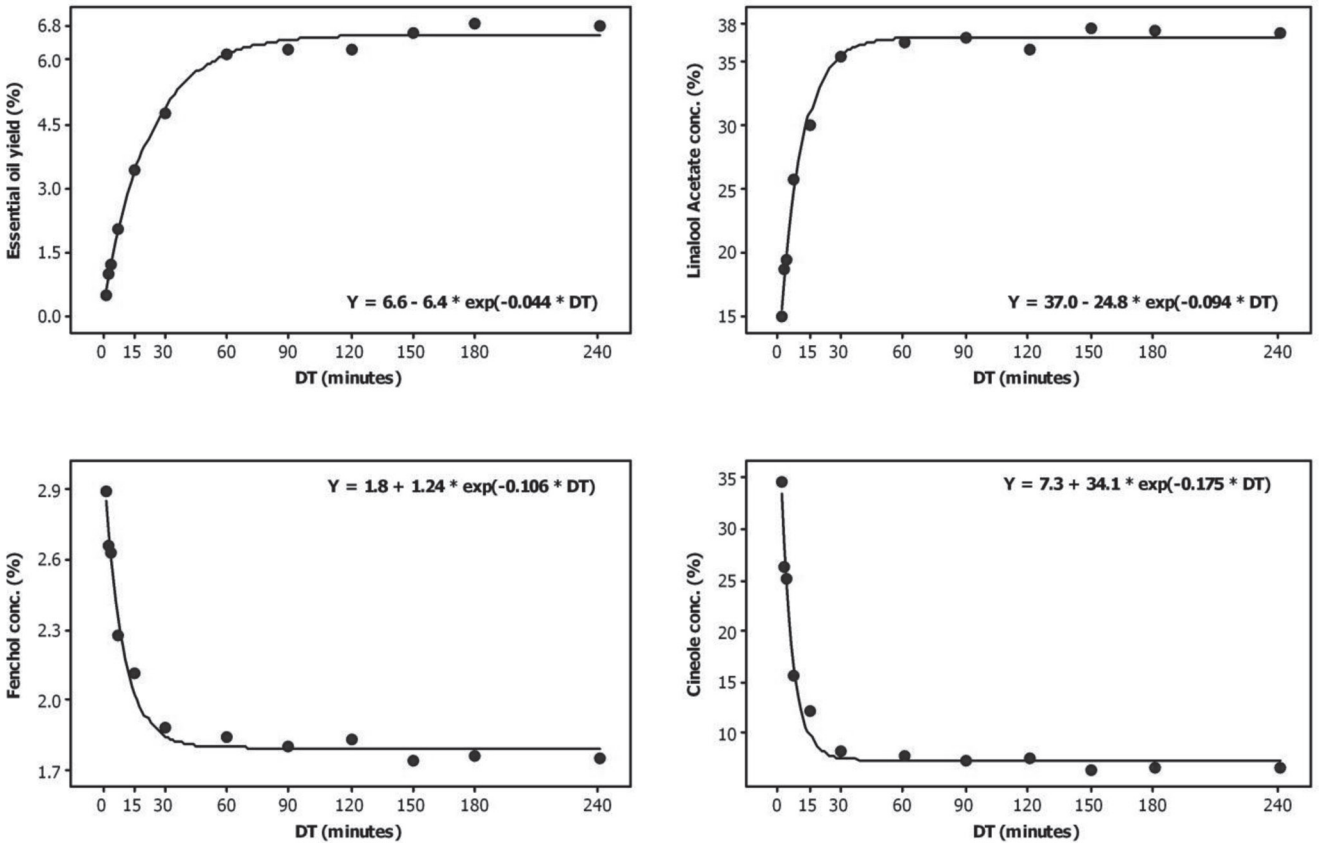

Fig. 2 Plot of Essential oil and the concentration of three constituents vs. distillation time (DT) along with the fitted (solid line) asymptotic regression model.

Table 1 Mean essential oil (EO) yield (wt/wt %), and concentration (total FID area %) of cineole, fenchol, camphor, and linalool acetate from 12 distillation times (DT).

DT (min)	EO yield	Cineole	Fenchol	Camphor	Linalool Acetate
1.5	0.50 e	34.52 a	2.89 a	6.57 f	15.0 c
3	0.97 e	26.21 b	2.66 b	7.78 cde	18.6 c
3.75	1.18 e	25.13 b	2.63 b	8.20 cd	19.4 c
7.5	2.02 d	15.57 c	2.28 c	9.06 ab	25.8 b
15	3.44 c	12.13 c	2.11 c	9.22 a	30.1 b
30	4.77 b	8.15 d	1.88 d	8.39 bc	35.4 a
60	6.14 a	7.61 d	1.84 d	7.66 de	36.5 a
90	6.25 a	7.12 d	1.80 d	7.64 de	37.0 a
120	6.26 a	7.45 d	1.83 d	7.49 e	36.1 a
150	6.64 a	6.37 d	1.74 d	7.20 ef	37.8 a
180	6.83 a	6.61 d	1.76 d	7.37 e	37.5 a
240	6.78 a	6.49 d	1.75 d	7.29 e	37.3 a

Within each column, means followed by the same letter are not significantly different at the 5% level of significance.

1.5 min DT, increased to reach maximum at 7.5-15 min DT, and then decreased to lower values at 60-240 min DT (Table 1). Camphor concentration at 30 min DT was not significantly different from the one at 7.5 min DT. The concentration of linalool acetate (range 15-38%) was the lowest at 1.5-3.75 DT, increased at 7.5-15 min DT, then increased again to reach a maximum at 30 min DT; further increase in DT did not alter its concentration (Table 1).

3.2 Nonlinear regression modeling results

The solid lines that represent the fitted nonlinear regression models shown in Fig. 2 suggest that the relationship between DT and essential oil yield as well as the concentration of cineole, fenchol and linalool acetate can be modeled by the asymptotic regression model (Eq. 1) almost perfectly. However, the asymptotes for EO yield and the concentration of linalool acetate was the maximum, whereas the asymptote for the concentration of cineole and fenchol was the minimum. The parameter estimates shown within each plot indicate that the maximum EO yield and linalool acetate concentration achievable were 6.6% and 37% respectively; on the other hand, the minimum cineole and fenchol concentrations that obtained from high DT were 1.8% and 7.3% respectively. These fitted models can also be used to predict essential oil yield and the concentration of cineole, fenchol and linalool acetate at any given DT.

4 DISCUSSION

In this study, lavender essential oil content reached 6.6%, which is higher than most previous reports[3, 13, 22, 23]. The reason is that in this study we utilized dried inflorescences, while in most of the previous reports the material distilled included stems and possibly some leaves. Since the essential oil glands are located in the flowers and not in the stems of the inflorescences[3], oil yield would be expected to be higher if only the flowers are distilled. Lavender composition in this study was comparable to the one reported previously[3, 13, 22, 23].

It has been known that lavender essential oil composition can be affected by a number of factors such as genetics (species or variety) or origin[13], environmental conditions such as fertilization, pesticide use, latitude[3], harvest time and drying[13].

Distillation time has been shown to have an effect on essential oil yield and composition of other crops such as peppermint, lemongrass, and palmarosa[14], oregano and pine[16, 17].

5 CONCLUSIONS

This is the first report to demonstrate that DT could significantly change lavender essential oil yield and composition. For optimal oil yields, dried lavender flowers should be distilled for 60 min. Lavender oil with a high concentration of cineole can be obtained at a very short DT. Alternatively, if high concentration of camphor is desirable, then lavender dry flowers should be distilled for 7.5-15 min.

Lavender oil with a high concentration of linalool acetate could be obtained when lavender dry flowers are distilled for at least 30 min. DT can be used as a simple tool for obtaining lavender oil with variable chemical profiles. This study also suggests that DT must be taken into consideration when comparing lavender essential oil yield and composition among different literature reports.

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