GC-MS Comparison of *Lavindin Grosso* Oil Obtained by Steam Distillation and SFE

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Abstract

Flowers like lavender has been used for scenting lotions , perfumes, and soaps dating back to the Egyptian era. Their essential oils have been used in applications as diverse as medicine and the culinary arts. The essential oil industry continues to grow with the popularity of aromatherapy; the market for quality lavender oil remains in demand.

Lavindin Grosso was harvested by hand at Peaceful Acres Lavender Farm in Martinsburg, OH. Oil was extracted by traditional steam distillation at Miami University Middletown and by supercritical fluid extraction with carbon dioxide at The Ohio State University in Susan Olesik's laboratory. Extracts were analyzed by GC/MS to compare the oils obtained and to compare them to commercially-available essential oils from France, Spain, and South Africa. No major differences were seen due to the extraction techniques; differences occurred due to the source of lavender. Oil yield will be determined with the next harvest. This work was performed to investigate a possible new agricultural endeavor in Ohio; SFE with carbon dioxide could be used to extract oils from several different crops.

Procedure

Extraction of the Essential Oil by Steam Distillation:

The fresh flowers were stored at room temperature in brown paper bags to prevent mold growth. The essential oil of one bunch was obtained by steam distillation on July 12, 2013. Distillation apparatus was set up according to normal procedure using a 3-neck, 1-L round bottom flask in a heating mantle with a cold-water condenser and a receiving vessel. A Powermite controller was used to adjust the temperature of the heating mantle.

Flowers were removed from the stalks by gently shaking the flower stalks in the paper bag and then manually pulling more off the stalks. Flowers (38.422 grams) were placed in the round bottom flask with 500 mL distilled water. The flask was heated to boiling, and the Powermite was adjusted to maintain a constant simmer for 2 hrs 25 minutes. The distillate (175 mL) was collected in a 400-mL beaker with a small visible oil layer. [Subsequent extractions used a separatory funnel to remove water while retaining the lighter oil layer.] Samples of aqueous distillate, droplets in the condenser, a thin, oily film on the distillate (when transferred to a tall, narrow container), and the "tea" remaining in the flask were stored in 2 mL amber vials for analysis. The smell of lavender was strong in all the samples.

Figure 1. Steam Distillation set up

By Supercritical Fluid Extraction (SFE):

The flowers were weighed and placed in the sample cylinder. To prevent clogging the capillary vent line with flower fragments, the flowers were placed in a silk bag inside the stainless steel cylinder. Initial trials used 1 gram of flowers; later extractions scaled up the process to 16 grams. Typically, the flowers were placed in the sample cylinder, which was then attached to the pump and the capillary vent. The pump reservoir was filled with 260 mL of liquid CO_2 at 56 bar (about 812 psi). This was pressurized to 80.0 bar (about 1160 psi) and then transferred to the sample cylinder, where the pressure was returned to 80.0 bar.

Figure 2. SFE set up (front view)

The cylinder was simultaneously heated to 35-50°C (warm to the touch) and covered with aluminum foil to trap the heat. The inlet from the pump was closed, and the sample was allowed to sit under pressure and heat to maintain supercritical conditions for the CO_2 for 30 minutes. The extract was removed from the cylinder by placing the capillary vent line into a conical test tube, opening the vent line, and carefully venting the CO_2. The extract was dissolved in the supercritical fluid but deposited in the test tube when the solvent became a gas. Fine control of the vent valve was needed to prevent depositing dry ice in the test tube or, at the other extreme, to prevent blowing the extract out of the test tube. The volume of oil extracted and captured was low, 0.1 mL per extraction. [0.1 mL from 16 grams of flowers means that 160 kg lavender flowers would be needed to extract 1 L oil. This is actually similar to the commercial steam distillation efficiency.] A further study was done to measure the completeness of the SFE process. Flowers (16.67g) were placed in a silk bag in the sample cylinder and were extracted with CO_2 at 80.0 bar and 50°C. During the venting process, some oil escaped the test tube and could be felt on the fingers holding the test tube. This extra virgin oil was colorless and weighed 0.0829g in the sample vial.

To determine the efficacy of the extraction, the sample was not removed from the sample cylinder. Instead the cylinder was charged with another 112 mL CO_2 at 80.0 bar, heated, and allowed to sit for 30 minutes. This time the extract (0.2425g including a drop of water) was yellow; 0.1626 g were transferred to a 2-mL amber vial. The same flowers were extracted a third time with 95 mL CO_2 at 80.0 bar for 34 minutes. The extract was pale yellow; 0.0819 g were transferred to an amber vial. The spent flowers were removed from the sample cylinder and discarded. For comparison to the flowers, the stalks (18.72 g) were finely chopped and extracted for 1 hr with the same procedure using 91 mL CO_2 at 80.0 bar and 50°C. Very little oil (0.0114 g) was extracted; 0.0069 g were transferred to an amber vial.

Analytical Conditions

Analysis of the Essential Oil: (GC-MS)

Parameters:

Column:	30 m x 0.25 mm	*Column Temperature Program:*	
Stationary Phase:	5% phenyl methyl	Initial Temperature:	40˚C
Carrier gas:	1.0 mL/min Helium	Initial Hold Time:	0.0 min
Split injection:	150:1	Rate:	2 ˚C/min
Sample Volume:	0.5 uL to 0.8 uL	Final Temperature:	210˚C
Injection mode:	Manual	Final Hold Time:	1 min
Inlet Temp:	250˚C	Total time:	86 min
Detector Temp:	280˚C	Mass Range Scanned:	10-450 m/z

Data/Results

Extracts were usually analyzed in duplicate; data are presented in Table 1 for the twelve most-concentrated components in the complex mixtures. Steam distillation was performed on several batches, as was SFE. The multiple extractions of one batch by SFE are designated SFE-1, SFE-2, and SFE-3 for three sequential extraction of the same flowers without removal of the flowers from the sample cylinder. The essential oils from South Africa, Senanque Abbey, Chateau du Bois, and "French" were commercial products.

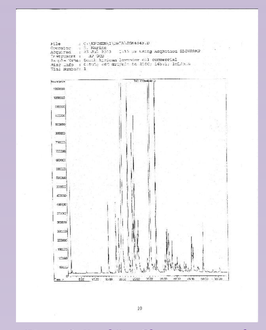

Figure 3. Total Ion Chromatogram of S. African Lavender Oil

Figure 3 shows a typical total ion chromatogram of the lavender oil from S. Africa.

Table 1: The 12 Most Concentrated Components of *Lavindin Grosso* Oil

CAS #	Ret Time (min)	Component	Area% SFE 7/19	Area% SFE-1	Area% SFE-2	Area% SFE-3	Area% Steam Distillate Fresh in duplicate / (moldy in duplicate)[1]	Area% S.Africa	Area% Senanque Abbey *(lanvandin grosso)*	Area% Chateau du Bois (fine oil; from *Hidcote*)	Area % French
470-82-6	15.2	Eucalyptol	16.6	19.3	17.0	11.6, 12.7	13.0, 13.2 / (23.14,21.6)	4.8, 0.9, 0.9, 0.73, 1.05	4.8	0.2	1.24,1.63, 1.67
3338-55-4 (cis) 3779-61-1 (trans)	15.9	1,3,6-octatriene, 3,7-dimethyl	4.7	8.2	4.8	ND, 2.8	6.6, 5.3 / (0.12, 0.08)	ND	0.7	7.6	ND
502-99-8	16.35	1,3,7-Octatriene, 3,7-dimethyl	1.5	2.1	1.1	9.0	2.1, 1.6 / (0.18, 0.11)	ND	0.2	3.8	ND
78-70-6	20.2	1,6-octadien-3-ol, 3,7-dimethyl (Linalool)	33.1	31.4	31.7	43.1, 42.8	16.7, 36.3 / (24.37,29.1)	34.7, 41.6, 29.36, 39.95, 17.82	36.2	18.6	35.7, 13.4, 16.4
76-22-2 or 464-49-3	22.5	Camphor or 1,7,7-trimethyl-bicyclo[2.2.1]heptan-2-one	5.5	5.3	6.0	4.6, 5.3	11.6, 5.0 / (12.03,8.96)	8.2, 7.9, 9.0,7.6,10.5	7.5	0.2	5.59,7.54, 7.56
507-70-0	24.1	Borneol	7.8	~4	9.7	7.9, 8.8	DNI[2](~15), 8.4 / (10.2, 16.51)	4.5, 4.6, 5.1, 4.5, 6.3	4.9	0.2	2.56, 3.46, 3.16
562-74-3	24.8	3-cyclohexen-1-ol, 4-methyl-1-(1-methylethyl)	3.5	2.8	1.1	2.9, 2.8	4.7, 3.4 / (ND[3], ND)	4.0, ~4.5, 0.89, 1.05	5.7	5.4	0.05, 1.62, 1.67
78-36-4 or 7149-26-0	30.3	Linalyl butyrate or 1,6-octadien-3-ol, 3,7-dimethyl propanoate	7.5	11.8	10.5	8.7, 9.0	14.4, 6.9 / (8.49, 7.08)	25.9, 36.8, 41.1, 35.2, 47.95	25.8	41.2	34.4, 51.6, 49.1
141-12-8	32.5	2,6-octadien-1-ol, 3,7-dimethyl-, acetate (Geraniol acetate)	1.2	1.5	1.5	1.3, 1.3	4.8, 1.1 / (2.87, 2.35)	3.1, 4.3, 5.03, 4.36, 6.1	3.3	5.6	5.13, 3.86, ND
87-44-5	40.0	Caryophyllene	0.8	0.9	1.0	0.9, 0.8	ND, 0.9 / (ND, ND)	ND	1.2	5.9	ND
18794-84-8	42.8	1,6,10-dodecatriene, 7,11-dimethyl-3-methylene	3.9	4.2	4.4	4.0, 3.8	5.0, 4.1 / (ND, 0.02)	ND	1.2	1.4	0.05, 0.16, ND
72691-24-8	55.59	α-Bisabolol	2.0	0.7	0.8	2.0, ~1.2	8.4, 2.1 / (ND, ND)	ND	0.1	ND	ND
		Total of above compounds	88.1	92.2	89.6	96.0, 91.3	87.3, 87.4 / (81.44, 85.81)	85.2, 96.1, 91.41, 92.31, 90.77	91.6	90.1	84.7, 79.9, 79.6

Notes:
1. A bundle of moldy flowers was also steam distilled. The essential oil had a woody smell, rather than the typical lavender aroma. It was analyzed in duplicate; the results are shown in parentheses.
2. DNI: Did Not Integrate, but the peak was present and the component was identified by MS.
3. ND: Not Detected; no peak was detected at that retention time that could be identified as the component of interest.

Conclusions and Future Work

Multiple analyses of the same extract demonstrated repeatability of the analytical method. Occasionally peak integration problems occurred, as noted in Table 1 with DNI, thus skewing the area% results. Extraction of the same crop of flowers on different days yielded oils of roughly the same composition, within the variability of duplicate analyses. All three sequential supercritical fluid extractions of lavender flowers produced oil of nearly the same composition. Some variability was seen but was within the variability of the analytical precision. The color differences are proof that minor variability does exist.

The difference in essential oil obtained by steam distillation and by SFE is inconclusive. The variability between batches of distillate bracketed the composition obtained by SFE. There is a distinct difference between the oil from *Lavindin Grosso* and the oil from *Lavindin Hidcote*. The differences among the *Lavindin Grosso* oils could be attributed more to where the lavender was grown than by the extraction method. The most common extraction method used today in the essential oil industry is steam distillation, which is probably the method used for the commercial lavender oils tested.

Future work will optimize the SFE conditions for lavender, measure the oil production yield, and investigate the feasibility of commercial application in Ohio.

References

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