Comparison of Super Critical Fluid Extraction and Hydrodistillation Methods on Lavander´s Essential Oil Composition and Yield

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Receive: 19 Feb. 2008 **Acceptance:** 8 Mar. 2008

Abstract

Background: **Lavender's essential oil is commonly used in aromatherapy and massage. Its major clinical benefits are on the central nervous system. Linalyl acetate and linalool are the most predominant chemical constituents in the essential oil of *Lavandula angustifolia*.**

Objective: **Comparison of super critical fluid extraction (SCFE) and hydrodistillation (HD) methods on Lavander´s essential oil composition and yield methods.**

Method: **In this work we extracted essential oil of this plant with two different methods; SCFE and HD and further analyzed by GC and GC-MS method.**

Results: **seventeen compounds were identified in the oil which prepared by HD, the major components of them were j. pinene (35.9%) and lavandulyl acetate (14.1%). In the SCFE extracted sample, the most frequent components were linalyl acetate (73.5%) and lavandulyl acetate (7.5%).**

Conclusion: **According to obvious difference in the composition of the essential oils prepared by two different methods (SCFE & HD), it seems that extraction method differ chemical composition of the oil and probably affects pharmacological properties.**

Keywords: **Lavender, Supercritical extraction, Hydrodistilation, Essential oil**

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[DOR: 20.1001.1.2717204.2008.7.25.23.9]

Introduction

The lavender essential oil has been traditionally obtained by steam distillation [1]. In the past few years, several new extraction procedures have been investigated as replacements for the traditional procedures [2]. Supercritical fluid extraction (SCFE) as an environmentally responsible and efficient extraction technique for solid materials was introduced and extensively studied for separation of active compounds from herbs [3]. Carbon dioxide (CO_2) is probably the most widely used supercritical fluid. Advantages include inertness, non-toxicity, non-explosiveness, and availability with high purity at low cost. Furthermore, relatively low critical properties make CO_2 (T_c=304.1 K, P_c=73.8 bar) an ideal solvent for the extraction of thermally labile components such as essential oils [4].

Lavender (*Lavandula angustifolia*) belong to the Labiatae family and have been used either dried or as an essential oil for centuries for a variety of therapeutic and cosmetic purposes [5]. Lavender is used in aromatherapy as a holistic relaxant and is said to have carminative and anticolic properties. Its sedative nature, on inhalation, has been shown both in animals and man. There is also an effect on certain brain waves such as the contingent negative variation [6].

Lavender oil, is chiefly composed of linalyl acetate, linalool, lavandulol, 1,8-cineole, lavandulyl acetate and camphor. Whole lavender oil and its major components linalyl acetate and linalool are used in aromatherapy, and in the flavoring and fragrance industries. Linalyl acetate has narcotic actions and linalool acts as a sedative [7]. Also lavender oil has been shown antidopaminergic, anticonvulsant and muscle relaxant effects in mice. While the exact cellular mechanism of action is unknown, one author has suggested that lavender may have a similar action to the benzodiazepines and to enhance the effects of gammaaminobutyric acid in the amygdale [8, 9]. Others have found that linalool inhibits acetylcholine release and alters ion channel function at the neuromuscular junction [8]. Its anti convulsive mechanism has been attributed to its competitive antagonistic effect on L-glutamate [9].

Previous studies on lavender extraction by SCFE demonstrate that the extract contains high levels of linalool and linalyl acetate [10, 11]. The high percentage of the latter compound is particularly important because the conventional steam distillation process leads to partial hydrolysis of linalyl acetate and thus lowers the value of the product [11]. Comparison of essential oils compositions obtained SCFE and HD have been reported for Ferula assa-foetida, Carum capticum and Salvia marzayanii plant [12, 13, 14].

In this work, chemical composition of *lavandula angustifolia* oil prepared by two methods hydro distillation technique and extraction by supercritical CO_2 have been reported. The yield of extraction for each method determined at the end.

Materials and methods

Plant Material

The aerial parts of *Lavandula angustifolia* were collected from medicinal plants research Institute of Shahid Beheshti University in June 2007. Immediately prior to SCFE the sample was ground in a blander to produce powder. Ethanol (99.6% purity) was purchased from Daru Pakhsh CO., Iran. CO_2 of high purity (>99.9%) was purchased from Daga Co., Iran. APO (Sigma, Germany) dissolved in 0.1% ascorbic acid.

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Supercritical CO_2 Extraction

A schematic diagram of the apparatus, which was used in this study, is shown in Fig. 1. Carbon dioxide is drawn from the source cylinder by a deep tube and after passing through a dryer is condensed by a cooling device. The CO_2 is then fed into the extractor through a particular double syringe pump. The extractor is a vessel of 0.4 liter internal volume, and is placed in an equipped oven for exact temperature regulation. The supercritical CO_2 leaves the extractor via a micrometering valve which controls the pressure discharge in the system.

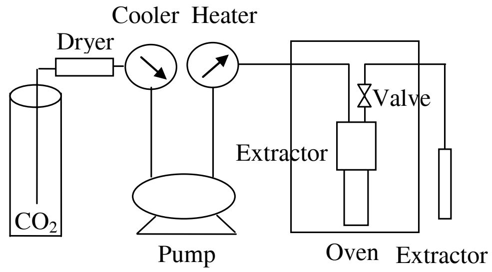

Fig. 1. Schematic diagram of supercritical extraction apparatus

A known quantity of lavender sample 20g was mixed with 5ml ethanol and 10g glass beads then was placed in the extractor. Before the start of extraction, the extractor was preheated in the oven for 60min. The extraction conditions were as follows: extraction time, static extraction for 90min and then dynamic extraction for 30min; temperature, 45^{o}C; pressure, 200bar; flow-rate of liquid carbon dioxide, 10ml/min. The extract was collected in a glass vial containing 4ml of ethanol.

Hydro distillation

The plant of dry material powder (100 g powder) was submitted to hydrodistillation for 5h, using a Clevenger-type apparatus (Shiraz, Iran), according to the USP Pharmacopoeia [12]. The volatile distillate was collected over normal pentane and refrigerated about one week till time of analysis and pharmacological tests. The yield of the oil was 2.5 % (v/w) based on dry plant weight.

GC and GC/MS Analysis

The chemical composition of the oil was investigated by means of chromatographic–spectrometric methods. The analytical gas chromatography was carried out using a Thermoquest 2000 GC chromatograph with capillary column DB-1 (30 m x 0.25 mm x 0.25 µm); carrier gas, He; split ratio, 1:25, and a flame ionization detector. The column temperature was programmed at 50°C for 1 min and then heated to 265°C with a 2.5°C/min rate and then kept constant at 265°C for 20 min. GC-MS was performed on a Thermoquest 2000 with a quadrupole detector, on capillary column DB-1 (see GC); Carrier gas, He; flow rate, 1.5ml/min and oven temperature as above. The MS operated at 70 eV ionization energy. Retention indices were calculated by using retention times of n-alkans that were injected after the oil at the same chromatographic conditions. The compounds were identified by comparison of retention indices (RRI) with those reported in the literature and by comparison of their mass spectra with the Wiley library [13] or with the published mass spectra [14].

Result and discussion

The hydrodistillation process has been traditionally used for the extraction of essential oils on a laboratory scale. In this study, use intends to the compare the efficiency of this process with its relationship to the volatile comparison the extracts from Lavender obtained by SCFE.

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The SCFE extracts and hydrodistillation of Lavender showed a relatively simple GC-MS chromatographic pattern. Detailed identification and quantization on the compounds found in Lavender oil produced by SCFE and HD were performed by GC-MS as reported in table 1, 2.

Table 1. Composition of the essential oil of *Lavandula angustifolia* extracted by SCFE

No	Compound	RI	%	KI
1	Linalyl acetate	43.46	73.53	1249.3
2	Lavandulyl acetate	45.32	7.47	1279.7
3	Trans-caryophyllene	52.05	3.69	1422.5
4	j-Farnesen	53.55	1.99	1459.8
5	Terpendiol	48.18	1.81	1340.4
6	Unknow	34.81	1.66	1079.1
7	Unknow	26.55	1.37	940.4
8	t-cadinene	54.6	0.91	1482.5

Table 2. Composition of the essential oil of *Lavandula angustifolia* extracted by HD

No	Compound	RI	%	KI
1	j-pinene	35.72	35.91	1009.2
2	Lavandulyl acetate	45.32	14.14	1306.8
3	Geranyl acetate	48.52	7.39	1380.0
4	Trans-caryophyllene	52.10	6.69	1466.0
5	Trans-ocimen	30.98	4.8	1021.0
6	Neryl acetate	47.92	3.89	1366.3
7	j-Farnesene	53.57	3.44	1502.0
8	j-Myreene	28.78	2.22	985.4
9	Linalyl acetate	44.31	2.21	1284.8
10	β-pinenoxide	40.35	2.18	1200.4
11	t-cadinene	54.62	1.42	1529.5
12	sabinaketon	35.01	1.30	1095.6
13	Limonen	29.61	1.12	997.9
14	Cis-Ocimen	30.44	0.95	1012.8
15	α-Terpinolene	31.87	0.79	1037.1
16	Linalool	39.41	0.71	1081.8
17	j-chamigren	51.62	0.53	1454.3

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In addition, the results as shown in table 3. for yield comparison. The major compounds lavender oil by hydrodistillation is: J-pinen, lavandulyl acetate, geranyl acetate and trance caryophellene.

Significant difference in the linalyl acetate content between the SCFE and the hydrodistillation production is notified from table 1, 2.

However, the recovery of linalyl acetate in SCFE is better than hydrodistillation. Finally SCFE show result in comparison with the conventional hydrodistillation procedure. Furthermore, SCFE gives a better selectivity for compounds of interest, changing extraction variables are less tedious, and it requires a shorter extraction time.

Table 2. indicate that the number of the essential oils components extracted by the SCFE (8 components) is lower than those obtained by the hydrodistillation method (17 components).

Comprehensive comparison of the lavender oils obtained by SFE and hydrodistillation: table 3.
color and texture are the prime characteristics and quality factors of essential oils, extraction yield and extraction time is the important parameter for the industrialization. Therefore, comprehensive comparison of the Lavender oils obtained by these methods was further listed in table 3. In table 1. the content of linalyl acetate and Lavandulyl acetate was determined by GC, SCFE offers the most important advantages over HD method. Extraction yield of SCFE was about six times as high as that obtained by hydrodistillation. Pale yellow oil is desired and shortest extraction time is needed for SCFE characteristics of the Lavender by SCFE and HD.

Table 3. **Comparison of SCFE and Hydrodistillation yield**

Method	yield	Extraction time	Color and texture
SCFE	12.5	5h	Pale yellow
Hydrodistillation	2.5	90min	Colorless

Conclusion

The supercritical fluid extraction of Lavender was studied, and the results, were compared with essential oil composition obtained by hydrodistillation. The SCFE method offers many important advantages over hydrodistillation. Extraction yield of SCFE was about six times as high as that obtained by hydrodistillation. Also, extraction time of SCFE is considered as the suitable process for obtaining Lavender oil with high quality on the hydrodistillation.

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