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Comparison of Microwave-Assisted Hydrodistillation and Traditional Hydrodistillation Methods for extraction of the *Vitex pseudo-negundo* essential oils

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ABSTRACT

Volatile compounds of Vitex pseudo-negundo samples were extracted by steam-hydrodistillation and microwave-assisted hydrodistillation. GC-MS analysis of the oils revealed the presence of 29 and 32 compounds in the essential oils obtained through normal and microwave-assisted method, respectively. The total yield of the volatile fractions obtained through nornal and microwave-assisted extraction was 1.11% and 1.56%, respectively. The two oils contained the same dominant components: α -Terpineol acetate (17.26% normal; 17.48% microwave), □-Himachalene (14.65% normal; 16.74% microwave), and Tetrahydro Rimuene (4.74% normal; 5.51% microwave).

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INTRODUCTION

Nowadays, essential oils are used as flavoring materials and in the preparation of perfumes and pharmaceutical products [1]. Besides the therapeutically application, the essential oil is widely applied in the cosmetic industry producing various bathing essences, hair lotions and shampoos and as a component of disinfectants and insecticides [2].

There are many methods to obtain essential oils from the plant materials [3]. The main methods to obtain essential oils from the plant materials are hydro-distillation, steam distillation, steam and water distillation. Hydro-distillation method suffers from several disadvantages including losses in the volatile compounds and long extraction time. In order to reduce the extraction time and possibly improve the extraction yield, to enhance the quality of the extracts and also to reduce the operation costs, new approaches such as microwave-assisted hydro-distillation. Microwave-assisted hydro-distillation is one way of using microwaves in an extraction process where a higher extraction rate (i.e. a shorter extraction time) along with a lower cost of operation can be obtained. The amount of essential oils obtained in 30 min with this method was comparable, both from qualitative and quantitative points of view, to those obtained after 4.5 h hydro-distillation [4].

Vitex is a genus of flowering plants in the family *Lamiaceae*. About 250 species are known in the world [5].The medicinal properties of this species have been long recognized [6]. *Vitex pseudo-negundo* is one of these medicinal herbs, and grows naturally in the vicinity of seasonal rivers in Iran [7]. The fruits were formerly used as a substitute for pepper from Italy to Eastern Georgia, a use which is still reflected [8]. It has long been used additional medicine for its action on the endocrine system [9]. A decoction of the roots has also been used as a febrifuge. The flowers have been prescribed with honey in fevers accompanied with vomiting and severe thirst. The fruits have been reported to be employed in amenorrhea [10]. Reduced body odor, reduced bloating ,reduced headache, reduced breast engorgement ,reduced restless leg syndrome, reduced arthritis and improved lubrication of eyes. disturbed sleep, rash or prick ling sensation, lethargy, sneezing/watery eyes, involuntary twitch (eyes), vertigo and headache. In other research GC-MS analysis of essential oil of dried leaves of Vitex pseudo-negundo identified 10 compounds of which 3 compounds were characterized as sesquiterpenes(47.14%) present in high amount; α-Copaene (25.26%), a sesquiterpene was the predominant constituent present in oil [11].

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The objectives of this study were to investigate the potential of microwave for the hydro-distillation extraction of essential oils from *V.pseudo-negundo* aerial parts to realize a comparison between microwave assisted hydro-distillation and normal hydro-distillation processes and chemical composition of essential oil extraction from *V. pseudo-negundo* by these two methods.

MATERIAL AND METHODS

Plant Material:

Thirty The aerial parts of *V. pseudo-negundo* growing in Bandar Abbas (Provincial capital of Hormozgan) were collected at the flowering stage in April, 2013. Aerial parts of plants were air-dried at room temperature (25°C) in the shade.

GC–MS:

GC/MS analysis was carried out by using Agilent technologies 7890 gas chromatograph with a mass detector Agilent technologies model 5975 C). The gas chromatograph was equipped with a HP-5MS capillary column (phenyl methyl siloxan, 30 m×0.25 mm i.d., Agilent technologies 19091S – 433 (60 to 325/350 ^{o}C). The oven temperature was programmed from 60 ^{o}C (0 min) to 220 ^{o}C at the rate of 5 ^{o}C/min and then hold for 10 min at 220 ^{o}C. Helium was selected as the carrier gas and flow rate was adjusted as 1 ml/min. The mass spectrometer (Agilent technologies 5975 C) was operating in EI mode at 70 eV. The mass range was 30–600 m/z; the interface temperature was 280 ^{OC}. Identification of components was based on a comparison of their KI and mass spectra with Willey (275) and Adams libraries spectra.

Hydrodistillation:

In conventional hydro-distillation, Clevenger apparatus was used. Heating was achieved using a hemisphere heater with 200 W of power. For the experiments,350 g of distilled water and 50 g of dried *V. pseudo-negundo* were placed in a flat bottom flask. The process was continued until no more essential oil was obtained. The essential oil samples were collected and stored in amber colored vials, dehydrated with anhydrous sodium sulfate, capped under nitrogen and kept at 4°C until being analyzed.

Microwave-assisted hydro-distillation:

A domestic microwave oven (Micro synth, Italy, 1000 W; variable in 110 W increments, 2.45 GHz) was modified for microwave-assisted hydro-distillation operation. 70 grams of *V. pseudo-negundo* was placed in a flask containing deionized water (1200 ml). The flask was setup within the microwave oven cavity and a condenser was used on the top (outside the oven) to collect the extracted essential oils. This period was sufficient to extract all the essential oils from the sample. At the end of extraction process, essential oils were weighed and stored in amber vials at 4 °C until they were used for analysis.

Results:

Chemical composition of the essential oil The chemical composition of the volatile fractions obtained from *V. pseudo-negundo* during the two methods extraction processes are represented together with the retention indices in Table 1. The GC–MS analysis of the oil samples revealed the presence of a total of 29 in normal hydro-distillation components and 32 in microwave-assisted hydro-distillation components. The total yield of the volatile fractions obtained through normal and microwave-assisted hydro-distillation was 1.11% and 1.56%, respectively.

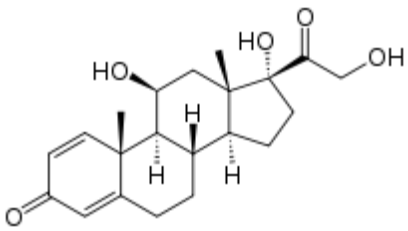
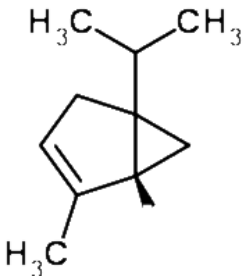
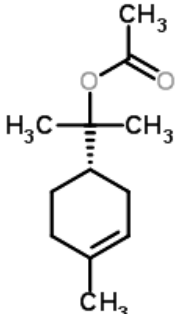
The two oils recovered from normal and microwave-assisted extraction contained the same dominant components: α -Terpineol acetate (17.26% Normal; 17.48% Microwave), δ-Himachalene (14.65% Normal; 16.74% microwave), and Tetrahydro Rimuene (4.74% Normal; 5.51% Microwave) respectively. These major compound are shown in Tablae 2 .

Kinetics of essential oil extraction from V. pseudo-negundo using microwave-assisted hydro-distillation compared with that of hydro-distillation. Extraction with microwave-assisted hydro-distillation started at a much earlier time than that with hydro-distillation (7 min vs. 30 min, respectively). This is due to the more efficient heating in the microwave system. Unlike the classical conductive heating methods, microwaves can heat the entire sample almost simultaneously and at a higher rate. Regarding the appearance, the color of the essential oil extracted by microwave-assisted hydro-distillation was somewhat lighter than that obtained by hydro-distillation. The ultimate yield of essential oils extracted by microwave-assisted hydro-distillation after 30 min of operation was greater than that obtained by hydro-distillation after 4.5 h of operation (1.1w/w , 1.56w/w).

Table 1: Chemical composition of *V.pseudo-negundo* essential oils obtained by hydrodistillation HD and microwave extraction MAHD.

No	Compound	KI	Hydrodistillation	Microwave	KI/MS
1	α-Thujene	930	29.10	---	KI/MS
2	α -Pinene	939	---	24.51	KI/MS
3	Sabinene	975	2.46	3.21	KI/MS
4	β -Pinene	979	0.54	0.46	KI/MS
5	β -Myrcene	991	1.36	1.03	KI/MS
6	α -Phellandrene	1003	0.73	0.62	KI/MS
7	p-Cymene	1025	0.6	0.53	KI/MS
8	Sylvestrene	1031	11.75	10.05	KI/MS
9	Isoterpinolene	1088	0.35	0.26	KI/MS
10	Linalol	1096	0.42	0.51	KI/MS
11	4-Terpineol	1177	0.34	---	KI/MS
12	α-Terpenol	1189	0.78	0.91	KI/MS
13	Bornyl acetate	1288	---	0.28	KI/MS
14	α -Terpineol acetate	1349	17.24	17.48	KI/MS
15	Sesquisabinene	1459	1.2	1.51	KI
16	Cumacrene	1472	0.37	0.78	KI
17	δ-Himachalene	1483	14.65	16.74	KI
18	δ-Curcumene	1483	0.67	0.77	KI
19	Bicyclogermacrene	1500	0.77	1.19	KI
20	Germacrene A	1509	0.30	0.40	KI
21	Sesquicineole	1516	---	0.29	KI
22	α -Sesquiphellandrene	1522	---	0.29	KI/MS
23	Caryophylene oxide	1583	0.01	0.21	KI/MS
24	α -Atlantol	1608	0.37	0.41	KI
25	tau.-Cadinol	1640	0.69	1.06	KI/MS
26	α -Eudesmol	1654	0.71	0.68	KI/MS
27	α -Bisabolol	1686	0.32	0.34	KI
28	Cubitene	1879	0.66	0.88	KI
29	epi-Laurenene	1901	0.46	0.56	KI
30	Beyerene	1932	0.89	1.02	KI/MS
31	Tetrahydro Rimuene	1961	4.74	5.51	KI
32	Cembrene A	1967	0.57	0.75	KI
33	Manoyl oxide	1987	---	0.32	KI/MS
34	Pseudo phytol	1988	1.87	2.17	KI
35	Manool	2057	---	0.29	KI/MS

Table 2: The major component in olis extracted by two methos

δ-Himachalene	α-Thujene	α-Terpineol acetate
		

Conclusions:

Microwave-assisted hydrodistillation is an advanced hydrodistillation method based on the use of a microwave oven. This method offered substantial advantages over conventional hydrodistillation. A similar extraction yield was achieved at significantly shorter extraction time when using microwave instead of conventional hydrodistillation. Microwave-assisted hydrodistillation can be suggested as a modern, fast and green extraction method.

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