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Research regarding obtaining volatile oils from native plants in microwave assisted vacuum systems

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Abstract

This study aims at examining the process of obtaining volatile oils through microwave hydro-distillation under vacuum. Using the classic hydro-distillation technique as a starting point, the authors have designed their own installation, activated with the help of microwaves. They have obtained as such volatile oils superior to those obtained through the classic technique.

Keywords: hydro distillation, microwaves, volatile oil, vacuum

1. Introduction

The World Health Organization based in Geneva launched programmes to increase the knowledge about plants used in traditional medicine. Being aware of the valuable Romanian achievements in the field of plant therapy, the United Nations Organization for Industrial Development (U.N.O.I.D), based in Vienna, have yearly organized classes in Bucharest, to train specialists in the field of plant therapy.
Studies and complex physical-chemical analysis have been conducted all over the world on volatile oils. As a result, a variety of options and systems are used in order to obtain volatile oils. These different options and systems rely on water steam techniques because of the high steam pressure which characterizes them. Amongst all the procedures used in order to obtain volatile oils, water steam transport is to date the most worldwide used method.

2. Problem Statement, background

Systematic research conducted years ago by using improved versions of the old water steam transport method, supercritical fluids extraction and microwave field processing followed by gas-chromatographic analysis have led to a series of very important observations. By corroborating these observations with older conclusions, we now know that a series of genuine compounds that exist in plants subjected to hydro-distillation are transformed, leading to the appearance of various artefacts in the oils. This is the reason why, starting from the same plant in different laboratories, or using devices with different features, volatile oils of inferior quality as well as oils with improved qualities can be obtained (as a consequence of artefact appearance) [1].

3. Paper approach

3.1. Methodology

Starting from the data presented in specialized literature regarding the process of obtaining volatile oils, the research team designed its own hydro-distillation system with a microwave assisted vacuum installation [2]. This study is a sequel of previous research team studies regarding microwave field process. Because of its complexity, this process requires integrated calculus and control systems for the microwave source, as well as the creation of parameter databases (treatment prescriptions).
The microwave exposure system consisted of a multimodal microwave oven with adjustable power between 700-900 W, in which a glass balloon, an external refrigerating system and an adjustable vacuum pump were inserted through structural modifications. The installation diagram is presented in Fig. 1

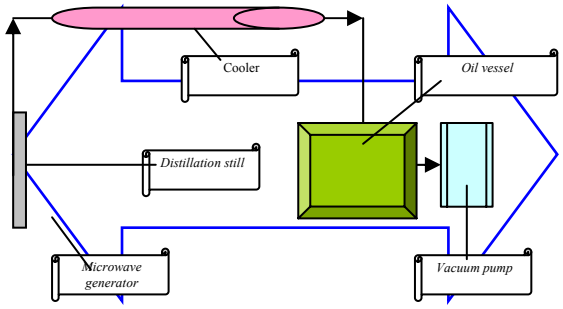

Fig. 1 Experimental installation

In order to compare the results from a qualitative and quantitative point of view, an old hydro-distillation system using a Neo-Clevenger on two types of plant material was created in parallel.

3.2. Experimental arrangement

The extraction of the volatile oils was carried out in the following manner:

- Extraction method: classic hydro-distillation using a Neo-Clevenger device and microwave assisted vacuum hydro-distillation designed installation;
- Plant material original status: fresh and dry;
- Season and geographic area: we used material from the Aromatic and Medicine Plant Research Laboratory test field – Brasov 2004-2005, specifically from the spontaneous vegetation of the Sibiu area, Romania;
- The collected plant material belonged to the following species: Menthae Folium – spearmint and Pinus Silvestris – pine;
- The classic hydro distillation parameters were: p = 1.00 atm., T = 100^0 C, contact time = 3.30 hours, distillate water, dry and grinded vegetal oil, grinded at φ = 0.15-0.25 mm;
- The microwave assisted vacuum hydro-distillation parameters were: p= 0.50-1.00 atm., T = 80-100 ^{0} C, contact time = 25 minutes, dry and grinded vegetal oil, grinded at φ = 0.15-0.25 mm.

3.3. Case study

At first, a microscopic histochemical examination was realised on transversal section of the vegetal material, to identify volatile oils in the plant tissue. These identifications were made based on specific volatile oils colour reactions [3]. R III SUDAN Solution Steimetz reactive with red colour was used for Menthae Folium vegetal material. Analytical data for volatile oils obtained with the two water steam carrying methods were made by using the Gas-Chromatographic-Mass Spectrometer (GC/MS) coupling technique.
In the case of gas-chromatographic analysis, the drips identification was made based on the standard retention times for the previous presented work conditions, and a determination of the quantitative composition was made based on the drips area.

3.4. Results& discussions

The efficiencies of the volatile oils obtained using both methods are presented in Table 1.

Exp. no.	Volatile oil	Efficiency of the hydro distillation process [%]	
		Microwave at 700W and 0.1MPa	Classic hydro distillation
1	SPEARMINT	1.20%	1%
2	PINE	0.7%	0.5%

Table 1 Volatile oils efficiencies

The gas-chromatographic and spectrometric analyses of the main compounds and the comparative analysis chart of the compounds obtained using both methods are shown, for spearmint in Table 2 and Fig. 2 and for pine in Table 3 and Fig. 3.

No.	Compound name	MINT 1 [% mass]	MINT 2 [% mass]
1	α-pinen	0.20	2.01
2	β-pinen	-	0.78
3	cis-p-Mentan-3-on	9.50	10.53
4	Menthol	67.91	61.45
5	Pulegone	0.30	2.16
6	Piperitone	8.95	3.44
7	Isomentol acetate	8.34	3.74
8	Burbanene	0.35	1.72
9	Allo-Aromadendren	-	6.19
10	Aromadendren	-	0.87
11	Germacren	-	0.67
12	Cadinen	-	0.57
13	p–mentan 8 -tiol	0.37	-
14	Carotol	0.17	-
15	Other compounds	3.92	5.87

Table 2 Results of the gas-chromatographic and spectrometric analysis for spearmint

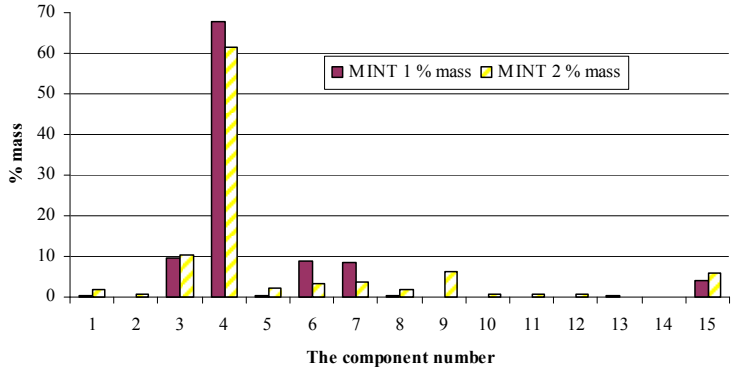

Fig. 2 The spearmint volatile oil components

No.	Compound name	Pine1 [% mass]	Pine2 [% mass]
1	α -pinen	17.61	8.25
2	Camfen	0.38	3.50
3	β -pinen	7.24	4.40
4	3 –Caren	7.70	-
5	α - limonen	14.46	10.27
6	γ - terpinen	0.22	0,19
7	Terpinolen	1.94	0.88
8	Terpenol	2.71	4.30
9	Acetat de bornil	4.91	18.39
10	Longipinen	1.67	-
11	α-Gurjunen	19.60	27.76
12	Thujopsen	7.21	3.63
13	Humulen	1.16	0.54
14	Aromadendren	0.76	0.25
15	α-Muurolen	1.12	0.80
16	δ-Cadinen	2.43	2.20
17	Caryofilen oxid	1.43	2.40
18	Juripen	0.75	2.25
19	Cembren	0.65	-
20	Other compounds	6.05	9.99

Table 3 Results of the gas-chromatographic analysis for pine

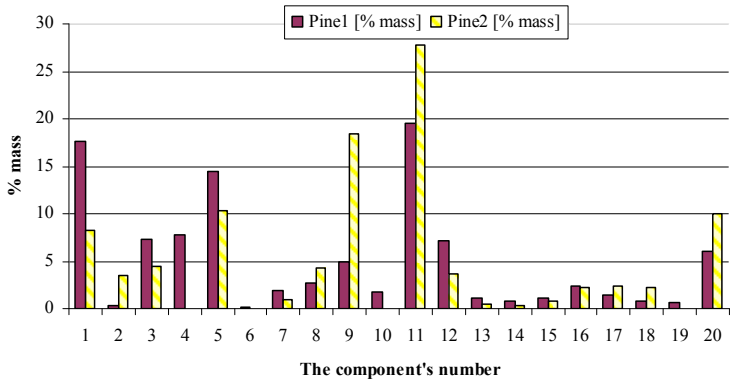

Fig. 3 The pine volatile oil components

The efficiencies for the experiment are presented in Table 4.

No.	**Obtained oil**	Efficiency η [%]	Power [W]	Pressure [MPa]
1	Spearmint	1.20	700	0.100
2	Spearmint	1.22	700	0.075
3	Spearmint	1.23	700	0.050

Table 4 Hydro distillation efficiency at constant power and different pressure

4. Conclusions

A higher percent of the main compounds which characterize volatile oils has been noticed when using the microwave hydro-distillation method. In the case of spearmint, this represents a menthol growth of about 10% and a significant piperitone growth – growth report of 2.6 and an izomenthol acetate growth of 2.22. An important fact is also the disappearance of p-menthan 8-tiol, product present only in the oil obtained with the help of microwaves. In the case of pine oil, this represents the double of the α -pinen quantity and a 40% growth of the α-limonen main compounds.
A remarkable fact is also the low content of certain secondary-artefact type of products when using the microwave assisted vacuum hydro-distillation method. This can be explained mainly because of the relative low hydro-distillation time (20 minutes only compared to the nearly 210 minutes in the case of classic hydro-distillation and lower temperature).
The future goals are to increase the obtained plant oil quality by changing the microwave extraction installations' work parameters (pressure and power). The design of a complex system for obtaining volatile oils by adding an ultrasound pre-treatment and post-treatment of the plant material subjected to hydro-distillation will also be taken into account.

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References

1. Ciulei, I., Grigorescu, E., Stanescu, U., Medicinal Plants, Plant Chemistry and Phytotherapy, 2nd vol., Medical Publishing House Bucharest, 1996, p 28-29;
2. Mosteanu, D., Research Regarding the Obtaining of Volatile Oils from Native Plants in an under Vacuum Microwave System, Doctor's degree thesis, Sibiu, 2005, p 109-110.
3. Marculescu, A., Extraction and Analysis of Active Elements out of Chrysantheum Balsamita, Doctor's degree paper, Faculty of Chemistry, Cluj-Napoca, 1994, p 41-42.