Comparison of extraction methods for the determination of essential oil content and composition of lavender leaves

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Abstract – ***In this paper, comparison of the volatile components composition in the samples obtained by hydrodistillation of Lavandula angustifolia leaves is described. The comparison of the lavender essential oil obtained by hydrodistillation in the Deryng and Clevenger type apparatus, according to the pharmacopoeial methods (FP VI and VII), showed the presence of the same terpenoids in both essential oils, however, the relative percentage composition of the components were different. The oils contain 37 identified compounds, of which the main are: borneol and caryophyllene oxide. Most of the compounds identified in the oils belong to monoterpenoids group (61.9-71.09%). The essential oils yields, measured after distillation according to Polish Pharmacopoeia VI (Deryng apparatus, 3 h) and VII (Clevenger type apparatus, 2 h), were the same and amounted to 0.45% v/w.***

Key words : *Lavandula angustifolia*, essential oil, hydro-distillation, Deryng apparatus, Clevengr-type apparatus.

I. Introduction

France is considered to be the homeland of lavender southern region. However, the lavender is also found in other European countries, including Poland. Currently, there are over 30 known species divided into six sections. Only two types among the *Lavandula* species are used for the production of essential oils on an industrial scale [1]. The percentage of individual constituent in the essential oil is variable and depends on genetic (species, chemical plant variety) and environmental factors (climate, insolation, altitude) [2]. Differences in the qualitative and quantitative essential oil composition can also be caused by the extraction procedure [3]. The predominant method used for the essential oils extraction is hydrodistillation, whereas the most modern techniques are solidphase microextraction (SPME) and microwaveassisted extraction (MAE) [4, 5]. In Poland, the predominant apparatus used for the essential oils hydrodistillation is Deryng apparatus. The Deryng apparatus was recommended for essential oils distillation by Polish pharmacopoeias till volume VI [6]. Polish Pharmacopoeia VII published in the 2006 [7] changed the hydrodistillation apparatus into Clevenger type. The Clevenger apparatus with its modifications is well known and used all over the world for many years. Figure 1 shows the differences in construction of Deryng and Clevenger type apparatus. The aim of this work is to compare the volatile components composition obtained from lavender leaves by hydrodistillation using Deryng and Clevenger type apparatus.

II. Experimental

Plant material

The material consisted of leaves 'Blue River' variety of lavender (*Lavandula angustifolia*). The plant was derived from experimental cultivation of Horticulture Department in West Pomeranian University of Technology in Szczecin from the set in July 2012. The plant samples were identified at the Department of Horticulture, Faculty of Environment Management and Agriculture of West Pomeranian University of Technology in Szczecin on the basis of voucher specimens data from Institute of Natural Fibres and Medicinal Plants, Poznan, Poland.

Hydrodestillation

Twenty grams of the powdered lavender leaves were submitted to water-distillation in the Clevenger type apparatus with 400 mL water for 2 h according to the Polish Pharmacopoeia VII [7]. The hydrodistillation was also performed using the Deryng apparatus with 400 mL water for 3 h according to the Polish Pharmacopoeia VI [6]. The essential oil yields were measured. Subsequently, the received essential oils were dried over anhydrous sodium sulfate and stored at 4°C until gas chromatographic determination of its composition.

GC/MS analyssis

The chemical composition of essential oils was determined by gas chromatography coupled to mass spectrometry (GC/MS) using an Agilent apparatus model 6890 with a chromatographic column HP-5MS of length 30 m, diameter 0.25 mm. Stationary phase film thickness was of 0.25 microns and the carrier gas was helium. Injector temperature was 250°C. A gradient of temperature was applied (60°C for 3 min., then an increase of 10 °C/min. to 300ºC). The qualitative analysis was performed by comparing MS spectra with the spectra of the NIST library. The identity of the compounds was confirmed by retention indices from literature data [8, 9]. Quantitative composition was determined by assuming that the sum of the individual compounds is 100%.

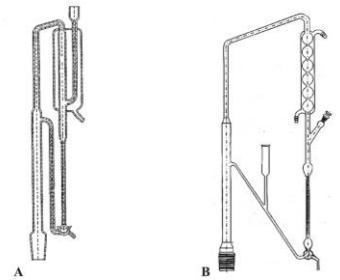

Fig. 1. Pharmacopoeias apparatus for hydrodistillation of essential oils. **A** n Deryng apparatus, **B** n Clevenger type apparatus [6, 7]

III. Results and discussion

The result of extraction methods *L. angustifolia* essential oil is presented in Table I. The composition of the essential oils of *Lavandula angustifolia* leaves obtained by hydrodistillation using Clevenger type and Deryng apparatus were investigated by means of GC-MS analysis. Altogether, 37 compounds were detected, which are listed in Table 1. The essential oils yields measured after distillation according to Polish Pharmacopoeia VI (Deryng apparatus, 3 h) and VII (Clevenger type apparatus, 2 h) were the same and amounted to 0.45% v/w.

TABLE 1

RELATIVE PERCENTAGE COMPOSITION OF THE ESSENTIAL OIL FROM LAVENDER LEAVES RECEIVED USING DIFFERENT EXTRACTION METHODS

Compounds	RI*	Distillation apparatus	
		Deryng	Clevenger type
α-pinene	932	1.28	1.95
camphene	946	0.74	0.90
3,7,7-trimetylo-1,3,5-cycloheptatriene	969	0.74	0.31
β-pinene	975	3.54	4.12
1-octen-3-ol	979	0.99	1.15
3-carene	1009	0.81	1.21
o-cymene	1021	1.53	1.95
p-cymene	1024	2.94	3.59
limonene	1028	2.25	3.15
eucalyptol	1031	6.19	8.50
γ-terpinene	1058	0.21	0.55
trans linalol oxide	1072	1.65	1.95
cis linalol oxide	1077	1.48	1.28
linalol	1100	4.90	5.12
octen-1-ol acetate	1111	0.75	0.75
nopinone	1137	0.41	0.21
camphor	1144	2.58	2.00
borneol	1168	13.84	15.21
4-terpineole	1178	0.90	0.90
p-cymen-8-ol	1184	1.83	1.22
crypton	1187	3.66	2.58
α-terpineole	1192	0.72	0.90
myrtenal	1196	2.23	2.55
eucarvone	1209	2.02	2.67
carveol	1221	0.32	0.21
borneol acetate	1228	1.05	1.55
cuminal	1242	1.28	0.50
carvone	1245	0.66	0.21
geraniol	1255	0.83	0.90
bornyl acetate	1285	0.53	0.21
lavandulol acetate	1289	1.37	1.89
geraniol acetate	1383	4.01	5.21
α-santalene	1420	2.63	2.95
α-bergamotene	1436	0.29	0.89
δ-cadinene	1516	2.00	2.34
caryophyllene oxide	1586	7.98	6.42
epibicyclosesquiphellandene	1645	6.82	6.12
Monoterpenoids	**61.90**		**71.09**
Sesqiuterpenoids	**19.72**		**18.72**
Other	**6.34**		**4.23**
Total [%]	**87.96**		**94.04**

European Pharmacopoeia V specifies the composition of lavender essential oil obtained from flowers *Lavandula angustifolia*, which is used for medicinal purposes as linalool contents should be in the range from 20 to 45%, linalyl esters from 25 to 46% [10, 11]. Our previous investigation showed that the highest content of linalool was in the 'Blue River' variety of 18.6% in flowers, and the lowest amounts in its leaves. In both varieties, the linalyl anthranilate was from12.8 to 7.7% in flowers, but was not found in leaves [12]. The chemical composition of essential oils obtained from the leaves, does not compatible with pharmacopoeial, significantly different the composition of the essential oil obtained from the flowers. However, the oils obtained from the leaves were characterized by a content of α-santalene, a chemical affecting antibacterial activity. An interesting compounds identified in the oils from the leaves of lavender is borneol, a compound belonging to the bicyclic mono-terpenes, with applications in medicine as an anesthetic Japanese [13].

Conclusion

In this paper the comparison of the lavender essential oil obtained in the Deryng and Clevenger type apparatus, according to the pharmacopoeial methods (FP VI and VII), was performed for the first time. In both essential oils the same compounds were detected, however, the relative percentage compositions of the essential oils components were different. The extension of extraction time, according to the method described in Polish Pharmacopoeia VI (Deryng, 3 h), caused the lost of highly volatile components such as monoterpenoids, but on the other hand, more sesquiterpenoids were present in the essential oil. The differences in relative percentage composition of the essential oils obtained using Clevenger type and Deryng apparatus can be explained by the differences in construction of both distillation apparatus and different extraction time.

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