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Influence of Hydrodistillation Conditions on Yield and Composition of Coriander (*Coriandrum sativum* L.) Essential Oil

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The yield and composition of essential oil obtained from the fruits of coriander (*Coriandrum sativum* L.) cultivated in Poland were investigated. Experiments were carried out by means of the hydrodistillation method using Clevenger and Deryng apparatus. No effect of the apparatus' construction on the essential oil yield was observed during a 2-h process (yield of 1.62%). The composition of the coriander essential oil was determined by using gas chromatography with mass spectrometry (GC-MS). The main components were linalool (78.45%), α-pinene (5.03%), camphor (3.90%), γ-terpinene (3.80%), D-limonene (2.58%) and geranyl acetate (2.13%). Moreover, the influence of different factors, such as time of hydrodistillation, sodium chloride addition and method modification by using microwave heating on essential oil yield, was studied. The addition of sodium chloride to distilled water did not affect the yield of essential oil, but slightly increased the content of camphor. Replacing conventional heating with microwaves during 15-min experiments enhanced the yield of essential oil from 1.20 to 1.35% and had a slightly greater influence on its composition, increasing the contents of some less volatile components: linalool, camphor, geraniol, *p*-cymene and D-limonene. The results obtained indicate that microwave heating may be a supporting method for the production of coriander essential oil using the hydrodistillation process.

INTRODUCTION

Coriander (*Coriandrum sativum* L.) is a member of the Apiaceae family and one of the earliest used spices in the world. This valuable medicinal herb comes from the Mediterranean region and nowadays is grown all over the world. Coriander fruits and leaves have been used as spices and traditional medicines since ancient times. Besides its attractive flavouring properties, the fruits have shown relaxant activity in the alimentary tract. The aroma of coriander leaves, which are traditionally eaten fresh, is completely different [Bhuiyan *et al*., 2009; Mandal & Mandal, 2015; Nurzyńska-Wiedrak, 2013; Sharma & Sharma, 2012]. Coriander fruits contain a great amount of biologically-active compounds and are the source of valuable essential oil. The coriander essential oil has a lot of uses in the food industry and medicine, as well as cosmetics applications. In the food industry, it is frequently used for its antibacterial, antifungal and antioxidant activities [Baratta *et al*., 1998; Begnami *et al*., 2010; Burdock & Carabin, 2009; Laribi *et al*., 2015; Mandal & Mandal, 2015; Sourmaghi, 2015]. Thanks to other interesting biological activities it exhibits, including hypoglycemic, hypolipidemic, anxiolytic, analgesic, anti-inflammatory, anti-convulsant and anti-cancer activities among others, this essential oil is a promising raw material for pharmacological and clinical applications [Laribi *et al*., 2015].

The main components of the coriander essential oil are oxygenated monoterpenes, with linalool being the dominant one. The rest of the characteristic constituents are α-pinene, β-pinene, β-myrcene, *p*-cymene, limonene, ɣ-terpinene, terpinolene, camphor and geranyl acetate. The chemical profiles of coriander seed essential oil significantly differ according to the origin of the plant material, due to the influence of genetic, climatic, seasonal and geographic conditions [Laribi *et al*., 2015]. Changes in the chemical composition of the coriander essential oil during the maturation process, with an increasing content of linalool, were also observed [Laribi *et al*., 2015; Mandal & Mandal, 2015; Msaada *et al*., 2007]. Moreover, fertilisation and weed infestation may also affect the chemical profile of the coriander essential oil [Gil *et al*., 2002].

The coriander essential oil can be obtained from coriander fruits using the hydrodistillation (HD) method. The aim of this study was to investigate the effect of different factors on the yield and composition of essential oil obtained by hydrodistillation. Method modifications included applying different hydrodistillation times, apparatus (Clevenger and Deryng), salting-out with sodium chloride (NaCl-HD) and microwave-assisted hydrodistillation (MAHD). The addition of various salts to distilled water during hydrodistillation can influence the composition of essential oil [Hassanpouraghdam *et al*., 2012]; however, the relevant available information is limited. In relation to the isolation of coriander essential oil, no data were found on the salting-out effect. According to the literature, the application of microwave radiation can effectively

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support the process of hydrodistillation; however, data related to the isolation of coriander essential oil are inconsistent [Kosar *et al*., 2005; Sourmaghi *et al*., 2015]. These facts encouraged us to undertake the study presented herein. The composition of the essential oil obtained from coriander cultivated in Poland was studied by gas chromatography with a mass selective detector.

MATERIALS AND METHODS

Materials and chemicals

Coriander fruits (*Coriandrum sativum* L.) were used as a source of essential oil. The plant material was obtained from Plant Herb "KAWON-HURT" Nowak sp.j. (Gostyń, Poland). According to producer information, the plant material was grown and harvested in Poland in 2015. Prior to essential oil isolation, coriander fruits were stored at room temperature in a dark glass container to protect them from light.

In order to identify the essential oil ingredients, the following standards were employed: camphene (≥90%), *p*-cymene (99%), R-(+)-limonene (97%), α-pinene (98%) and γ-terpinene (97%) from Sigma-Aldrich (Steinheim, Germany); camphor (natural pure) from International Enzymes Limited (Windsor, Berkshire, England); and β-pinene (≥95%) from Fluka (Buchs, Switzerland). Saturated *n*-alkanes standard C_7–C_{30}, purchased from Supelco (Bellefonte, Pennsylvania, USA) was applied to determine the linear retention indices of essential oil components.

The other reagents were anhydrous sodium sulfate (pure p.a.) from POCH (Gliwice, Poland) and acetone (pure p.a.) from Chempur (Piekary Śląskie, Poland).

Isolation of the essential oil

The samples of coriander fruits were subjected to hydrodistillation using an all glass Clevenger apparatus (WPL Gliwice, Poland) recommended by Polish *Pharmacopoeia*, 7th edition [2006] onwards, and a Deryng apparatus (WPL Gliwice, Poland) recommended by previous editions of Polish *Pharmacopoeia* [2002]. The fruits were ground immediately before the experiments in order to minimise essential oil losses. In order to extract the essential oil, 30 g of ground fruits, 500 mL of distilled water and some boiling stones were placed in a 1000 mL round-bottom flask. The flask was then connected to the apparatus and heated with an electric heating bowl to the boiling point. Hydrodistillation was performed at a rate of 3 mL/min for different periods of time ranging from 15 min to 2 h. The process of coriander essential oil isolation was also carried out using 500 mL of a sodium chloride solution (3%) instead of distilled water and with the application of a Samsung ME73E microwave oven (Samsung Electronics, Suwon, Korea) as a source of energy for heating (600 W). The essential oil yield was calculated on the basis of essential oil volume and plant material weight, and expressed in % (v/w). The obtained essential oils were dried over anhydrous sodium sulfate and stored in sealed vials at 4°C until GC-MS analysis was performed.

GC-MS analysis

The GC-MS analysis of the essential oil was carried out using an Agilent 6890 gas chromatograph (Agilent Technologies, Palo Alto, California, USA) equipped with an Agilent 5973 Network Mass Selective Detector (MSD). The separation was effected using an HP-5MSI capillary column with a bonded (5% phenyl)-methylpolysiloxane stationary phase (30 m × 0.25 mm I.D., 0.25 μm film thickness). The GC oven temperature was programmed from 50°C to 290°C at a rate of 4°C/min. The total running time for one sample was 60 min. Helium was used as a carrier gas at a constant flow rate of 1.2 mL/min. The injection port temperature was 250°C and 2 μL of solution was injected in the split mode at a ratio of 10:1. The mass selective detector conditions were as follows: electron impact positive ionization: 70 eV; full scan mode: mass range from 20 to 500 m/z; MSD transfer line temperature: 310°C; MS quad: 150°C; MS source: 230°C. The mass selective detector was turned off during the elution time of the solvent (0–3.00 min).

Prior to analysis, the samples of essential oil were dissolved in acetone. For this purpose, 1.0 mL of acetone was placed into a chromatographic vial (1.8 mL) and 20 μL of essential oil were added. The obtained concentration of essential oil solution was approximately 17 mg/mL. The vial was closed with a screw cap with a silicone/PTFE membrane, and mixed well. Three analytical samples were prepared for each examined essential oil.

The components of the essential oils were identified by comparison of their mass spectra with those collected in the NIST 02 mass spectral library. The identification was confirmed by linear retention indices determined for all components of the essential oils. The obtained results were compared with published data [Adams, 2007; Babushok *et al*., 2011]. The linear retention indices (LRI) were calculated for all volatile constituents using the retention data of a homologous series of *n*-alkanes (C_7–C_{30}) obtained under the same chromatographic conditions used for the analysis of the essential oils.

The quantitative analysis was performed using the internal normalisation method without the application of correction factors. The relative percentage of the essential oil constituents was evaluated from the total ion chromatogram (TIC) using the MestReNova 10.0.2 software. The relative contents of particular compounds in essential oils were obtained as the percentages of a peak area in a total ion chromatogram.

Statistical analysis

The results obtained from three separate tests were averaged and expressed as a mean ± standard deviation. The means were compared using one-way analysis of variance (ANOVA), followed by Duncan's multiple range tests at the significance level of 0.05 using Statistica ver.12 software [StatSoft, 2014].

RESULTS AND DISCUSSION

Essential oil was isolated from plant material cultivated in Poland. In the first stage, the influence of apparatus construction on the essential oil yield was studied. For this purpose, essential oil was obtained using two types of all glass apparatus – Deryng and Clevenger. The process of essential oil isolation was carried out for 2 h. The hydrodistillation

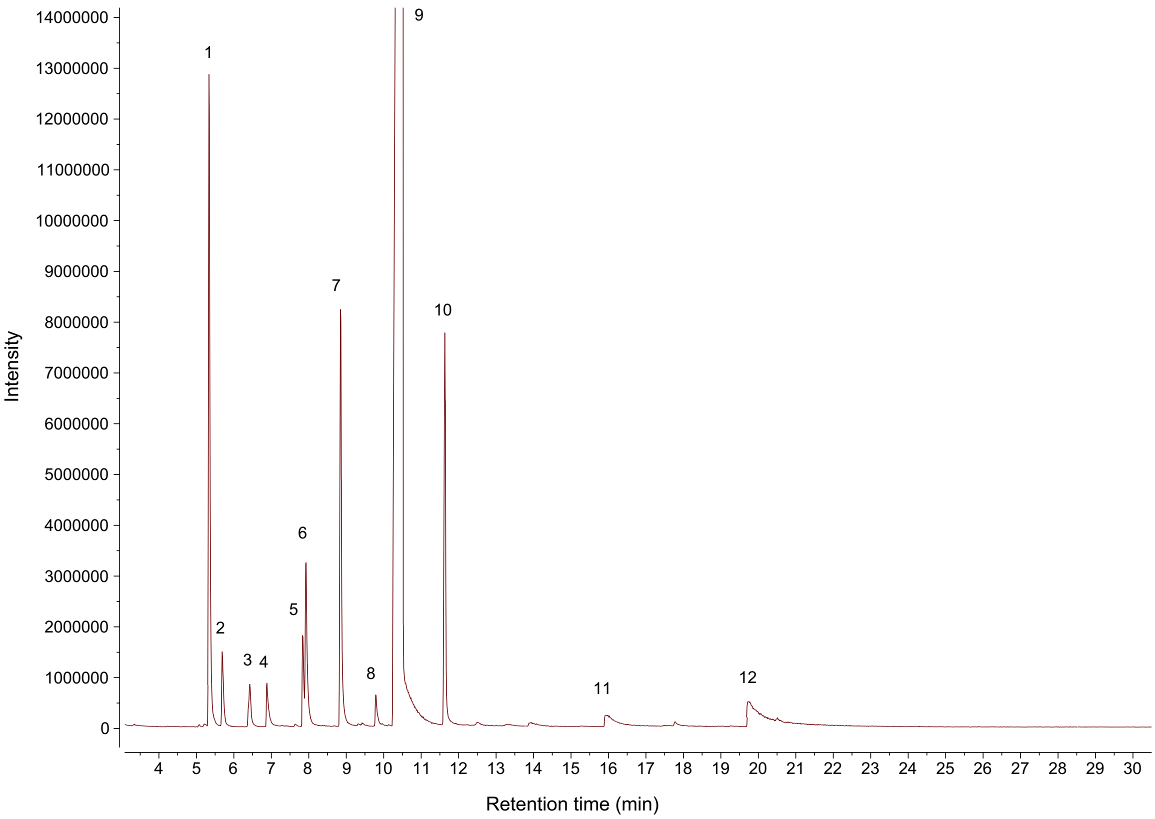

FIGURE 1. GC-MS chromatogram of the coriander essential oil obtained by a 2-h hydrodistillation (the number of peaks is consistent with data in Table 1).

of coriander fruits yielded a colorless liquid with a spicy citrus aroma. The obtained results indicate that the construction of the apparatus used had no influence on essential oil yield. The values were 1.61±0.09% (v/w) and 1.63±0.08% (v/w) for the Deryng and Clevenger apparatus, respectively. Statistical analysis showed that the obtained results were not different at $p>0.05$.

The coriander essential oil yield reported by other authors varies widely. In most cases, the coriander fruits contained between 0.10 and 0.60% of essential oil [Anwar *et al.*, 2011; Bhuiyan *et al.*, 2009; Ebrahimi *et al.*, 2010; Eikani *et al.*, 2007; Grosso et al., 2008; Msaada *et al.*, 2007; Pavlić *et al.*, 2015; Ravi *et al.*, 2017; Telci *et al.*, 2006; Verma, 2014]. Few authors reported higher values of coriander essential oil yield, ranging between 0.65 and 2.20% [Gil *et al.*, 2002; Zheljazkov *et al.*, 2008]. The results obtained in our study correspond with those reported by Gil *et al.* [2002] for plant material of European genotype. According to these authors, the fruits of this genotype have low average weight (~5 mg per dry fruit) and high essential oil content (from 1.34 to 1.62% (v/w)). The average weight of coriander fruits examined in our study was a bit higher – 7.68 mg per dry fruit but the content of essential oil was similar – on average 1.62% (v/w). Large-fruit types of *Coriandrum sativum* (~11 mg per dry fruit) grown in tropical and subtropical climates generally contain low amounts of essential oil (up to 0.5%) [Gil *et al.*, 2002; Telci *et al.*, 2006], but small-fruit types may also produce lower amounts of oil (0.37–0.43%) if they are cultivated in subtropical regions, *e.g.* in Turkey [Telci *et al.*, 2006]. Taking into consideration our results and reports in the literature, it can be stated that climate conditions in Poland may favour the cultivation of plant material for the production of coriander essential oil.

Using the GC-MS method, 12 components of coriander essential oil were identified. A typical chromatogram showing the separation of coriander essential oil constituents is presented in Figure 1. The determined linear retention indices of the particular compounds collected in Table 1 were consistent with the literature [Adams, 2007; Babushok *et al.*, 2011]. Moreover, the retention times of 7 compounds were confirmed by the analysis of standards. The presence of the identified compounds in the analysed oils was also confirmed by comparing their mass spectra with mass spectra from the NIST 02 library. A spectral search quality for substances present in the samples at high concentrations was above 90%, and for minor constituents above 80%.

As shown in Table 1, the main component of coriander essential oil was linalool, which is in agreement with the literature. The relative content of this compound was 78.45% of all detected compounds, which may indicate the final stage of coriander fruits' development [Msaada *et al.*, 2009; Verma, 2014]. Linalool belongs to oxygenated monoterpene hydrocarbons. The other compounds from this group were camphor (3.90%), geranyl acetate (2.13%), and geraniol (1.07%). The sum of oxygenated monoterpene hydrocarbons made up 85.55% of the essential oil. The monoterpene hydrocarbons, including α-pinene, camphene, β-pinene, β-myrcene, D-limonene, ɣ-terpinene and terpinolene, were the second largest class of determined substances. The content of this

TABLE 1. Composition of coriander essential oil obtained by a 2-h hydrodistillation.

No.	Component	LRI[1]	LRI[2]	LRI[3]	Identification[4]	Relative content[5] ± SD[6] (%)
1	α-Pinene	931	932	936±8	RT, RI, MS	5.03±0.10
2	Camphene	945	946	950±7	RT, RI, MS	0.64±0.01
3	β-Pinene	974	974	978±7	RT, RI, MS	0.48±0.03
4	β-Myrcene	991	988	989±6	RI, MS	0.47±0.03
5	*p*-Cymene	1025	1020	1024±7	RT, RI, MS	0.54±0.03
6	D-Limonene	1027	1024	1030±7	RT, RI, MS	2.58±0.06
7	ɣ-Terpinene	1058	1054	1060±8	RT, RI, MS	3.80±0.05
8	Terpinolene	1088	1086	1087±8	RI, MS	0.63±0.07
9	Linalool	1105	1095	1099±7	RI, MS	78.45±0.54
10	Camphor	1147	1141	1143±8	RT, RI, MS	3.90±0.07
11	Geraniol	1264	1249	1255±9	RI, MS	1.07±0.03
12	Geranyl acetate	1386	1379	1380±9	RI, MS	2.13±0.25

[1] LRI: linear retention indices relative to C_7–C_{30} *n*-alkanes on the HP-5MSI capillary column; [2] LRI: linear retention indices from literature [Adams, 2007]; [3] LRI: linear retention indices from literature [Babushok *et al*., 2011]; [4] RT: comparison of retention time with standard, RI: retention index, MS: mass spectrum; [5] Relative content: percentage of the peak area in the total ion chromatogram; [6] SD: standard deviation calculated from three separate tests.

group of compounds in essential oil was 13.63% with significant participation of α-pinene (5.03%), ɣ-terpinene (3.80%) and D-limonene (2.58%). The relative contents of other monoterpenes were below 1%. The presence of a small amount of cymene (0.54%), which is an aromatic hydrocarbon, was also detected. The sum of identified compounds represented 99.71% of the obtained coriander essential oil. Unlike previous reports [Anwar *et al*., 2011; Bhuiyan *et al*., 2009; Ebrahimi *et al*., 2010; Grosso *et al*., 2008; Telci *et al*., 2006; Verma, 2014], sesquiterpenes, aliphatic hydrocarbons and their oxygenated forms were not detected.

In the next stage of our research, the kinetics of hydrodistillation was studied. The yield of essential oil isolation was determined for different periods of time: 15, 30, 60, 90, and 120 min (Figure 2). Leading the process of coriander essential oil isolation for 15 min allowed obtaining the yield of 1.20±0.05% (v/w). Prolonged distillation resulted in a significant increase of the yield to a value of 1.38±0.11% (v/w) for 30 min and 1.56±0.07% (v/w) for 60 min. Longer hydrodistillation times (90 and 120 min) seem to be unreasonable from an economical point of view – the obtained differences in yields were insignificant (p>0.05).

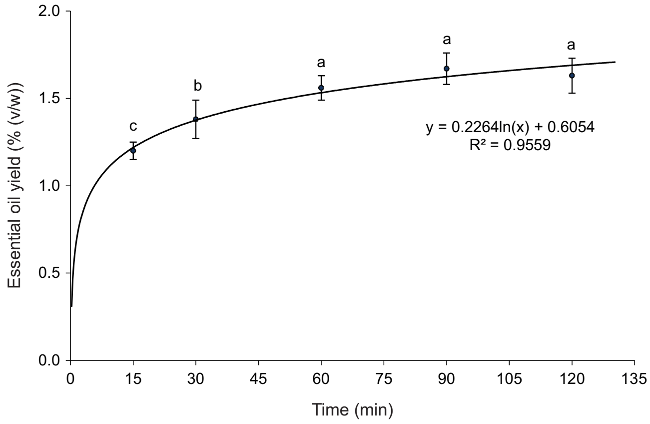

FIGURE 2. Influence of hydrodistillation time on the coriander essential oil yield. Values marked with the same letter are not significantly different (Duncan's test, p>0.05).

Taking into account the kinetics of the coriander essential oil isolation, 15-min experiments were performed to study the influence of other factors on the yield and composition of coriander essential oil. Essential oil obtained using the hydrodistillation (HD) process was labelled as a control. Applied modifications of the process included replacement of distilled water with a 3% sodium chloride solution (NaCl-HD) and the use of microwave radiation (MAHD) as a source of energy for heating. For these experiments, the Deryng apparatus was applied, as it was more convenient to use with the microwave oven.

The values of yields obtained at standard (HD) and modified conditions (NaCl-HD and MAHD) are presented in Figure 3. There was no significant difference (p>0.05) between the control and the hydrodistillation process with a sodium chloride solution. In both cases, the essential oil yields were 1.20% (v/w). Similar observations were made by Hassanpouraghdam *et al*. [2012], who investigated the effect of different salts on the efficiency of isolation and composition of essential oil from *Mentha pulegium* L. In contrast, the application of microwave radiation caused increase of the yield to 1.35% (v/w). This allows the shortening of the time for receiving the essential oil and reduces the costs of energy. In previous reports [Kosar *et al*., 2005; Sourmaghi *et al*., 2015], authors described the effect of microwave heating on coriander essential oil composition and activity. Unfortunately, it is difficult to compare the yields obtained in the reported experiments using the HD and MAHD methods, due

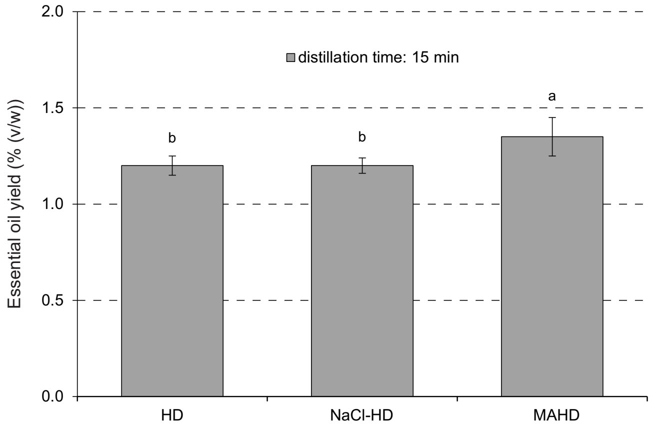

FIGURE 3. Influence of modification process on the coriander essential oil yield (HD: hydrodistillation; NaCl-HD: hydrodistillation with sodium chloride; MAHD: microwave-assisted hydrodistillation). Values marked with the same letter are not significantly different (Duncan's test, p>0.05).

TABLE 2. Effects of sodium chloride and microwave heating on composition of coriander essential oil obtained by a 15-min hydrodistillation.

No.	Component	Relative content[1] ± SD (%)		
		HD[2]	NaCl-HD[3]	MAHD[4]
1	α-Pinene	6.05 ± 0.21^{a}	5.57 ± 0.20^{b}	4.06 ± 0.23^{c}
2	Camphene	0.73 ± 0.04^{a}	0.70 ± 0.02^{a}	0.60 ± 0.03^{b}
3	β-Pinene	0.52 ± 0.04^{a}	0.53 ± 0.01^{a}	0.49 ± 0.03^{a}
4	β-Myrcene	$0.48\pm0.03^{a,b}$	0.52 ± 0.03^{a}	0.47 ± 0.02^{b}
5	*p*-Cymene	0.61 ± 0.02^{b}	0.61 ± 0.01^{b}	0.99 ± 0.02^{a}
6	D-Limonene	2.73 ± 0.05^{b}	2.65 ± 0.05^{b}	3.10 ± 0.09^{a}
7	ɣ-Terpinene	4.12 ± 0.09^{a}	3.89 ± 0.10^{b}	3.01 ± 0.11^{c}
8	Terpinolene	0.39 ± 0.01^{a}	0.33 ± 0.04^{b}	$0.38\pm0.01^{a,b}$
9	Linalool	77.10 ± 0.39^{b}	$77.65\pm0.34^{a,b}$	78.30 ± 0.37^{a}
10	Camphor	3.65 ± 0.02^{b}	3.88 ± 0.08^{a}	3.91 ± 0.02^{a}
11	Geraniol	0.70 ± 0.10^{b}	0.81 ± 0.09^{b}	1.06 ± 0.13^{a}
12	Geranyl acetate	2.59 ± 0.17^{a}	2.52 ± 0.32^{a}	3.04 ± 0.39^{a}

[1] mean values along the same lines with a different letter (a–c) are significantly different ($p<0.05$); [2] HD: hydrodistillation; [3] NaCl-HD: hydrodistillation with sodium chloride; [4] MAHD: microwave-assisted hydrodistillation.

to the different times of distillation applied. In studies performed by Kosar *et al*. [2005], however, higher yield of essential oil was obtained using microwaves for 50 min (0.5%) than using the standard hydrodistillation process performed for 3 h (0.4%).

As can be seen in Table 2, experiments using sodium chloride (NaCl-HD) and microwave heating (MAHD) led to obtaining essential oil with slightly different contents of some components compared to the control (HD). It can be noticed that the addition of sodium chloride slightly decreased the content of α-pinene (from 6.05 to 5.57%), γ-terpinene (from 4.12 to 3.89%) and terpinolene (from 0.39 to 0.33%). The contents of some less volatile components increased: camphor (from 3.65 to 3.88%) and linalool (from 77.10 to 77.65%); however, changes in linalool content were not significant. It is known from the literature that the influence of salting-out on oil composition during hydrodistillation can be meaningful, but this effect is both salt- and constituent-dependent [Hassanpouraghdam *et al*., 2012]. Information on NaCl treatment during the hydrodistillation of coriander essential oil was not found. In our research, changes in the composition of coriander essential oil due to the addition of sodium chloride to distilled water were generally not significant. Lower content of γ-terpinene obtained in an experiment with NaCl compared to the control (HD) was in agreement with results obtained by Hassanpouraghdam *et al*. [2012] for *Mentha pulegium* L. essential oil.

The application of microwave heating during hydrodistillation had slightly greater influence on essential oil composition, with the same tendency observed in increasing the contents of some less-volatile components and decreasing that of the most volatile ones. In the literature, there is some contradictory information related to the influence of microwaves on the content of linalool, which is the main component of coriander essential oil [Kosar *et al*., 2005; Sourmaghi *et al*., 2015]. In our MAHD experiment, the relative content of linalool was higher by 1.2% in comparison to the hydrodistillation carried out with conventional heating. Slightly higher contents of camphor, geraniol, *p*-cymene and D-limonene were also obtained. In contrast, the content of the most volatile compound α-pinene decreased by about 2%. Slightly lower contents of other volatiles (camphene and γ-terpinene) were also observed. Our studies indicate that using microwave heating during hydrodistillation may be a promising method for coriander essential oil production due to enhancing its yield in a shorter period of time.

CONCLUSIONS

On the basis of the performed experiments, it was found that a hydrodistillation time of 60 min is sufficient to achieve rewarding yields of coriander essential oil. No effect of apparatus construction (Clevenger and Deryng) on the yield was observed. The tested plant material was rich in essential oil – 1.62% (v/w). Analysis using the GC-MS method allowed the identification of 12 components, which accounted for more than 99% of the obtained product. The main components of coriander essential oil were: linalool (almost 80%), α-pinene, ɣ-terpinene, camphor, limonene, and geranyl acetate.

The addition of sodium chloride did not affect the yield of essential oil, but slightly increased the content of the main less volatile compounds – camphor and linalool; however, changes in linalool content were not significant. The amounts of more volatile components slightly decreased. Applying microwave heating during a 15-min hydrodistillation enhanced the yield of essential oil from 1.20 to 1.35% (v/w) and had a slightly greater influence on its composition. Increasing amounts of some less volatile components and decreasing amounts of more volatile ones were also observed. It seems reasonable to focus on the further development of the microwave-assisted hydrodistillation process for the isolation of coriander essential oil. Using this method allows for the reduction of time, costs of energy consumption, and obtaining products with similar or slightly modified composition in comparison to the conventional hydrodistillation method.

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CONFLICT OF INTEREST

The authors declare no conflict of interest.

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