Microwave-assisted extraction of essential oil from ginger (*Zingiber officinale* Rosc.)

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Comparison between conventional and microwave-assisted hydrodistillation at different power levels has been applied for the extraction of essential oil from *Zingiber officinale* Rosc. rhizome. In addition to the collected essential oils, as the main products, hydrolats were also collected, as valuable by-products from both extraction techniques. A comparison of two applied techniques was done in terms of extraction time, extraction yield, the chemical composition of essential oils and hydrolats, and environmental impact. Microwave-assisted hydrodistillation achieved a higher extraction yield (1.70, 1.70, and 1.85 % for power levels of 180, 360, and 600 W, respectively) compared to hydrodistillation (1.50 %). Furthermore, distillation time related to the energy consumption has been reduced from 144 min and 1.44 kWh for conventional hydrodistillation to 37, 32, 27 min and 0.11, 0.19, 0.27 kWh for power levels of 180, 360, and 600 W, respectively. Content of α-zingiberene in essential oil and hydrolat obtained by hydrodistillation was 29.89 and 6.87 %, while content of α-zingiberene in essential oils and hydrolats obtained by microwave-assisted hydrodistillation was higher yielding the amounts of 34.12, 34.43, and 42.00 % and 18.70, 22.60, and 32.92 % for power levels of 180, 360, and 600 W, respectively. Microwave-assisted hydrodistillation has proven to be promising technique for the isolation of ginger's essential oil regarding improved yield, reduced time and energy requirements, as well CO_2 emissions while maintains oil quality.

Key words: hydrosol, essential oil, volatile compounds, ginger, HD, MAHD

1. INTRODUCTION

Ginger (*Zingiber officinale* Rosc., Zingiberaceae) is a perennial plant species whose tuberous rhizome is widely used as a very popular spice, for food and beverages flavoring and as a medicinal raw material. The plant is native to Southeast Asia and India, but nowadays it is extensively cultivated due to its commercial importance (Kamaliroosta et al., 2013). Whole or cut, dried ginger rhizomes (*Zingiberis rhizoma*) with a minimum essential oil content of 1.5 % (v/w) are official herbal drug according to the European Pharmacopoeia 10.0 (Ph.Eur. 10.0., 2019). Ginger-contained medicines are traditionally used in various gastrointestinal disorders such as loss of appetite, dyspeptic ailments, stomach ache and diarrhea. In rational phytotherapy, as a confirmed safe antiemetic, it is primarily used in the prevention and treatment of postoperative nausea and vomiting, as well as kinetosis (Kamaliroosta et al., 2013; Tóth et al., 2018).

Chemically, about 400 identified ginger ingredients are generally divided into a group of pungent constituents and a group of aromatic constituents. Non-volatile oily compounds (gingerols, shogaols, zingerons, ginger) that contribute to the sweet-spicy taste of ginger are pungent constituents (Kamaliroosta et al., 2013; Talebi et al., 2021). The unique fragrance of ginger comes from essential oil. Its yield and composition depend on the origin of plant, plant genotype, maturity, harvest time, drying and extraction techniques (Ravi Kiran et al., 2013). The main ingredients of the essential oil are sesquiterpene hydrocarbons α-zingiberene, β-sesquiphellandrene, β-bisabolene, α-farnesene, *ar*-curcumene, and lower content of the monoterpene hydrocarbons and oxygenated monoterpenes (camphene, geraniol, citral, cineol, β-phellandrene) (Kamaliroosta et al., 2013; Talebi et al., 2021). Conventional hydrodistillation (HD) and steam distillation are still the most commonly used technique for essential oil isolation at both laboratory and industrial levels (Drinić et al., 2020). The main limitations of these traditional distillation techniques are the thermal and/or hydrolytic degradation of esters and polyunsaturated ingredients during prolonged exposure to elevated

Table 1. Chemical constituents in the *Z. officinale* rhizome essential oil obtained with hydrodistillation (HD) and microwave-assisted hydrodistillation (MAHD) at different power levels

#	Compound	RI[a]	HD	MAHD		
				180 W	360 W	600 W
1	2-heptanol	899.1	-	0.04	0.08	-
2	tricyclene	927.9	-	0.04	0.08	-
3	α-pinene	938.7	2.14	1.78	1.39	0.62
4	camphene	955.5	8.61	7.13	5.93	3.02
5	sabinene	978.5	-	0.03	0.06	0.12
6	β-pinene	982.8	0.27	0.21	0.22	-
7	6-methyl-5-hepten-2-one	985.7	0.76	0.35	0.27	0.08
8	myrcene	991.9	1.00	0.91	0.83	0.50
9	α-phellandrene	1009.0	0.25	0.24	0.24	0.16
10	β-phellandrene	1036.3	6.85	6.15	5.95	5.85
11	1,8-cineole	1037.8	3.52	3.14	3.80	1.15
12	terpinolene	1093.4	0.20	0.17	0.21	0.16
13	6-camphenone	1098.7	-	-	0.07	-
14	linalool	1101.2	0.37	0.40	0.46	0.40
15	citronellal	1154.7	0.19	0.20	0.22	0.19
16	camphene hydrate	1160.5	-	-	0.07	-
17	*iso*-chrysanthenol	1168.0	-	0.15	0.11	0.08
18	borneol	1176.4	1.22	1.39	1.61	1.53
19	*trans*-isocitral	1184.7	-	0.07	0.11	-
20	terpinen-4-ol	1187.6	-	0.11	0.13	0.12
21	α-terpineol	1198.5	0.59	0.67	0.77	0.73
22	myrtenol	1207.1	-	0.08	0.14	0.13
23	citronellol	1230.1	0.18	0.23	0.25	0.24
24	neral	1246.5	3.17	3.06	3.00	2.68
25	geraniol	1256.8	0.27	0.23	0.21	0.17
26	*iso*-chrysanthenyl acetate	1264.8	-	0.12	0.12	0.12
27	geranial	1275.3	4.12	4.13	4.15	4.16
28	bornyl acetate	1294.7	0.25	0.30	0.34	0.29
29	δ-elemene	1351.3	-	0.10	0.10	0.10
30	citronellyl acetate	1354.4	-	0.08	0.09	0.08
31	neryl acetate	1383.3	0.39	0.37	0.43	0.23
32	geranyl acetate	1385.8	0.16	0.18	0.16	0.29
33	β-cubebene	1392.3	0.30	0.31	0.32	0.33
34	*cis*-caryophyllene	1405.9	0.51	0.55	0.61	0.62
35	*α-cis*-bergamotene	1413.9	0.14	0.15	0.19	0.18
36	α-himachalene	1449.5	0.21	0.23	0.24	0.29
37	sesquisabinene	1461.3	0.32	0.32	0.28	0.24
38	*trans*-9-*epi*-caryophyllene	1467.1	0.27	0.33	0.38	0.53
39	γ-himachalene	1483.5	0.18	0.20	0.20	0.21
40	*ar*-curcumene	1494.7	4.44	3.10	2.87	2.41
41	germacrene D	1503.0	1.38	1.97	2.69	1.89
42	α-zingiberene	1510.6	29.89	34.12	34.43	42.00
43	β-bisabolene	1515.2	3.90	5.15	6.92	7.38
44	γ-cadinene	1518.7	0.95	1.12	1.41	0.45
45	7-*epi*-α-selinene	1522.7	6.71	2.23	-	-
46	β-sesquiphellandrene	1540.3	11.16	11.67	11.48	12.89
47	*trans*-γ-bisabolene	1546.0	0.32	0.40	0.47	0.58
48	longipinanol	1567.4	0.78	0.98	0.06	1.46
49	*trans*-nerolidol	1570.9	0.39	0.77	1.31	-
50	germacrene D-4-ol	1604.1	-	-	-	0.09
51	*trans*-sesquisabinene	1604.6	0.61	0.63	0.63	0.71
52	viridiflorol	1628.6	0.78	0.80	0.89	0.98
53	1-*epi*-cubenol	1645.5	0.55	0.57	0.60	0.70
54	allo-aromadendrene epoxide	1656.4	0.36	0.37	0.39	0.47
55	β-eudesmol	1679.1	0.44	0.52	0.62	0.74
56	*epi*-α-bisabolol	1703.1	0.90	0.97	0.93	-
57	2-*trans*-6-cis-farnesal	1716.2	-	0.12	0.24	1.12
58	2-*cis*-6-*trans*-farnesol	1727.9	-	0.15	0.06	0.29
59	*cis*-nuciferol	1748.9	-	0.10	0.09	0.11
60	2-*trans*-6-*trans*-farnesal	1754.1	-	0.11	0.11	0.14
	Non-oxygenated monoterpenes		19.33	16.62	14.84	10.43
	Oxygenated monoterpenes		14.43	14.91	16.22	12.59
	Non-oxygenated sesquiterpenes		60.68	61.95	62.58	70.10
	Oxygenated sesquiterpenes		4.80	6.09	5.93	6.80
	Other		0.76	0.43	0.43	0.08

[a] RI, retention indices as determined on HP-5 column using homologous series of C_8-C_{30} alkanes.

temperature in an aqueous medium. Loss of volatile compounds may also contribute to reduced extraction efficiency of the essential oil. Furthermore, traditional techniques as time and energy demanding are unfavorable for modern industry that aspires to establish economically and ecologically friendly extractions techniques (Ferhat et al., 2007). Considering these facts, the development of innovative green extraction techniques to overcome the drawbacks of traditional techniques is necessary.

In the last decade, microwave-assisted hydrodistillation (MAHD) has been affirmed as an effective, energy- and time-saving extraction technique for essential oils isolation (Drinić et al., 2020; Golmakani and Rezaei, 2008). Significant acceleration of the extraction process was achieved via rapid microwave heating and energy transfer generated by the electromagnetic field. Actually, traditional heating arises by thermal energy transferring from the heating surface, across extraction medium, along the temperature gradient. In a contrast of microwave heating, where the energy transmission via electromagnetic radiation is contactless and instantaneous. The electromagnetic radiation induces reorientation of dipole molecules (dipole rotation) and electrophoretic ion movement (ionic conduction). This generates friction between the molecules, creates kinetic energy and consequently induces a rapid increase in temperature. The MAHD technique is applicable in the laboratory and on an industrial scale (Radivojac et al., 2020; Vinatoru et al., 2017).

The main objective of this research was to compare the qualitative and quantitative composition of *Zingiberis rhizoma* essential oils and hydrolats obtained using traditional HD and novel MAHD methods. Essential oils and hydrolats were analyzed by gas chromatography-mass spectrometry (GC-MS). Additional objectives of the research were to compare time and energy consumption, as well as the impact of applied methods on the environment.

2. MATERIALS AND METHODS

2.1. Standards and reagents

Ethanol, distilled water, and *n*-hexane were purchased from Zorka Pharma, Šabac (Serbia). Sodium sulfate anhydrous (Na$_2$SO$_4$) was purchased from Sigma Chemicals Co. (USA).

2.2. Plant material

Zingiber officinale Rosc. rhizome was obtained by local market. Fresh plant material was cut and air dried at ambient temperature to residual humidity lower than 5 %. Before extraction process, plant material was ground in a domestic blender (CISA Cedacería Industrial, Barcelona, Spain).

2.3. Essential oils, hydrolats

Conventional HD and MAHD techniques were applied for the isolation of essential oils and hydrolats. HD was performed using a Clevenger type apparatus according to the procedure I of the Yugoslavian Pharmacopoeia IV (Ph.Jug.IV, 1951). MAHD was performed using the specially created system, consisting of the microwave oven (HMT 72M450, Bosch, Gerlingen, Germany) connected to the Clevenger type apparatus. The water/plant ratio was the same for HD and MAHD, 10:1 (w/w). MAHD was performed on three different power levels (180, 360, and 600 W) until no more essential oil was obtained (20 min). The time required to reach the final amount of essential oil, i.e., time required for starting the extraction and time for essential oil isolation, present a total extraction time. The essential oils were collected, and dried over anhydrous sodium sulphate. The essential oil yield, expressed as a percentage, was calculated on a moisture-free basis. Oil samples (20 μL)

were dissolved in 96 % ethanol (2 mL) and stored at 4 °C until further analysis.

Hydrolats were collected from the burette after HD and MAHD, extracted with n-hexane (5 mL of hydrolats with 3 × 1 mL *n*-hexane), hexane fractions were collected, combined and stored in the freezer until further analysis.

2.4. Chemical analysis of essential oil and hydrolats

The chemical composition of the essential oils and hydrolats was analyzed using GC/MS technique. GC/MS analyses were performed on a Shimadzu GCMS-QP2010 ultra mass spectrometer fitted with a flame ionic detector and coupled with a GC2010 gas chromatograph. The InertCap5 capillary column (60.0 m × 0.25 mm × 0.25 μm) was used for separation. Helium (He), at a split ratio of 1:5 and a linear velocity of 35.2 cm/s was used as carrier gas. Initially, the oven temperature was 60 °C, which was held for 4 min, then increased to 280 °C at a rate of 4 °C/min, and held for 10 min. The injector and detector temperatures were 250 °C and 300 °C, respectively. The ion source temperature was 200 °C. The identification of the constituents was performed by comparing their mass spectra and retention indices (RIs) with those obtained from authentic samples and/or listed in the NIST/Wiley mass-spectra libraries, using different types of searches (PBM/NIST/AMDIS) and available literature data (Adams, 2007; Hochmuth, 2006).

2.5. Environmental impact of applied techniques

Regarding environmental impact, electrical consumption and CO$_2$ emission were calculated. The electrical consumption (A) for HD and MAHD was calculated as the electrical power for a time, using the following equation:

$$A = P \times t,$$

where A is electrical consumption (kWh), P is electrical power (kW) and t is time (h). According to Ferhat et al. (2006) to obtain 1 kWh from coal or fuel, 800 g of CO$_2$ will be emitted in the atmosphere during the combustion of fossil fuel. CO$_2$ emission can be described by the equation:

$$E_{CO_2} = \frac{A \times 800}{1000},$$

where E$_{CO_2}$ is CO$_2$ emission (kg) and A is electric consumption (kWh).

3. RESULTS AND DISCUSSION

3.1. Essential oil yield

The essential oil for MAHD at power level 180 and 360 W was same and yielded 1.70 %, while at power level 600 W it was slightly higher and yielded 1.85 %. The yield of essential oil of the HD method was 1.5 %, and lower compared to the method of MAHD extraction. Similar to our results, (Govindarajan and Connell, 1983) reported that yield of ginger essential oil ranged from 1.0 to 3.0 %. Total extraction time was 37, 32, and 27 min for MAHD at 180, 360, and 600 W, respectively, while for HD it was 144 min. Time required to start the extraction was 17, 12, 7, and 24 min for MAHD at 180, 360, and 600 W, and HD, respectively. The extraction time was reduced by about 70-80 % using microwaves. High reduction in the processing time is probably due to different mechanism of heating in MAHD and HD. Namely, the heating process in HD is conditioned by the transfer of heat from the heating medium to the interior of the samples, while the microwave heating is volumetrically dispersing within the sample and is based on ionic conduction and dipole rotation (Veggi et al., 2012). As microwaves interact with the free water molecules present in the gland and vascular systems, local heating occurs

Table 2. Chemical constituents in the *Z. officinale* rhizome hydrolat obtained by hydro-distillation (HD) and microwave-assisted hydro-distillation (MAHD) at different power levels

#	Compound	RI[a]	HD	MAHD		
				180 W	360 W	600 W
1	hexanal	799.4	2.05	-	-	-
2	2-heptanol	898.9	3.19	2.89	1.21	-
3	*α*-pinene	938.9	-	-	0.71	0.50
4	camphene	954.8	1.69	2.75	3.30	2.52
5	6-methyl-5-hepten-2-one	986.1	12.26	5.70	2.10	-
6	*α*-phellandrene	1035.5	-	0.37	3.24	3.14
7	1,8-cineole	1037.4	27.62	21.21	16.41	8.57
8	linalool	1101.6	4.68	3.12	2.50	1.45
9	camphor	1154.6	-	-	0.69	-
10	borneol	1176.5	17.30	15.20	10.00	6.35
11	terpinen-4-ol	1186.2	-	-	0.72	-
12	*α*-terpineol	1198.7	11.76	9.15	6.14	3.58
13	neral	1245.6	1.46	3.45	3.81	3.67
14	geraniol	1256.7	2.50	1.05	0.89	0.54
15	*cis*-chrysanthenyl acetate	1262.4	2.15	1.82	1.01	0.58
16	geranial	1274.1	1.67	3.46	4.63	5.06
17	*ar*-curcumene	1493.6	-	2.56	3.04	4.32
18	germacrene D	1502.7	-	0.85	0.99	1.37
19	*α*-zingiberene	1505.9	6.87	18.70	22.60	32.92
20	*trans, trans-α*-farnesene	1513.0	-	-	2.52	3.49
21	*7-epi-α*-selinene	1518.2	-	-	-	1.00
22	*α*-bisabolene	1520.2	1.74	3.14	5.53	6.73
23	*β*-sesquiphellandrene	1537.3	3.05	4.58	7.96	11.42
24	longipinanol	1567.3	-	-	-	0.96
25	*trans*-sesquisabinene hydrate	1604.6	-	-	-	0.84
26	*α*-bisabolol	1628.5	-	-	-	0.97

[a] RI, retention indices as determined on HP-5 column using homologous series of C_8-C_{30} alkanes.

resulting in cell expansion and rupture allowing the easier isolation of the essential oil.

The difference in time required to start the extraction time at different power levels are correlated to the different density of the waves. The lowest used microwave power has the lowest density waves and therefore it has the longest time required to start extraction. Total extraction time was much lower in MAHD than HD even at the lowest microwave power (180 W). Reduction in the extraction time of MAHD compared to HD was previously reported (Ferhat et al., 2007; Golmakani and Rezaei, 2008; Rezvanpanah et al., 2008).

3.2. Composition of essential oils and hydrolats

The composition of the essential oil of ginger rhizome obtained by MAHD and conventional HD method is given in Table 1. The chemical composition of the essential oils obtained by both applied methods were found to be almost the same qualitatively, whereas some quantitative differences were observed. In the HD-obtained essential oil 43 compounds were found, while the MAHD-obtained essential oils contained 57, 58, and 51 compounds for power level 180, 360, and 600 W, respectively. The dominant compound in all samples was *α*-zingiberene (29.9-42.0 %) followed by camphene (3.0-8.6 %), *β*-phellandrene (5.8-6.8 %), and 1,8-cineole (1.1-3.5 %). Our findings are in accordance with previously reported data (Kamaliroosta et al., 2013; Ravi Kiran et al., 2013; Stoyanova et al., 2006). Content of *α*-zingiberene was higher in essential oils obtained by MAHD (34.12, 34.43, and 42 % for MAHD at 180, 360, and 600 W, respectively compared to 29.89 % for HD). The content of other major compounds was approximately the same for HD and MAHD, except for camphene, 1,8-cineole and *ar*-curcumene for HD and MAHD at 600 W, where the content of these compounds was about two-fold higher in HD, as well as for *β*-bisabolene whose content was about two-fold higher in the MAHD. About 80 % of the identified com-

pounds belong to the group of non-oxygenated terpenes, most of which (60-70 % of total oil) belong to sesquiterepenes, while a smaller part (10-19 % of total oil) belongs to monoterpene hydrocarbons. The amount of non-oxygenated compounds in essential oil obtained by HD and MAHD at 180, 360, and 600 W was 80.01, 78.57, 77.42, and 80.53 %, respectively, of which the sesquiterpenes take share 60.68, 61.59, 62.58 and 70.10 %. The content of oxygenated components was about 20 % in all analyzed samples, where the portion of monoterpene fraction was two to three times higher than the portion of sesquiterpene fraction. Although, many authors reported that essential oil obtained by MAHD contains higher amount of oxygenate compounds comparing to the HD (Drinić et al., 2020; Ferhat et al., 2006; Filly et al., 2014), we cannot confirm that pattern based on our results. Similar to our results, Karakaya et al. (2014) reported that the concentration of oxygenated and non-oxygenated compounds was almost the same in both methods, MAHD and HD.

The major components in essential oils were also the major components in hydrolats with different percent in different hydrolats samples (Table 2). The hydrolats obtained by HD and MAHD at 360 W had 1,8-cineole as dominant compound with relative percentage of 27.62 and 21.21, respectively, followed by borneol (17.30 and 15.20 %), *α*-zingiberene (6.87 and 18.70 %), *α*-terpineol (11.76 and 9.15 %), linalool (4.68 and 3.12 %), *β*-sesquiphellandrene (3.05 and 4.58 %), geranial (1.67 and 3.46 %), and *β*-bisabolene (1.74 and 3.14 %). The most abundant compound in hydrolats obtained by MAHD at 360 and 600 W was *α*-zingiberene with relative percentage of 22.60 and 32.92, respectively, followed by 1,8-cineole (16.41 and 8.57 %), borneol (10.00 and 6.35 %), *β*-sesquiphellandrene (7.96 and 11.42 %), *β*-bisabolene (5.53 and 6.73 %), *α*-terpineol (6.14 and 3.58 %), geranial (4.63 and 5.06 %), and linalool (2.50 and 1.45 %). Same trend, that the major compounds in essential oil were also major compounds in hydrolats, was reported previously

Table 3. Process parameters of microwave-assisted hydrodistillation (MAHD) compared with standard hydrodistillation (HD)

Distillation method	Process time	Time saving	Energy consumption	Energy saving	CO_2 emission	CO_2 emission reduction
	[min]	[%][a]	[kWh]	[%]	[kg]	[%]
HD (600W)	144	n/a	1.44	n/a	1.15	n/a
MAHD 180 W	37	74.3	0.11	93.19	0.09	92.28
MAHD 360 W	32	77.7	0.19	88.22	0.15	86.65
MAHD 600 W	27	81.2	0.27	84.75	0.22	81.19

[a] n/a, stands for not applicable.

(Paolini et al., 2008; Shahani et al., 2011). Obtained hydrolats can be considered for use in cosmetic and pharmaceutical industries due to valuable chemical constituents.

3.3. Environmental impact

Concerning environmental impact, the advantages of obtained essential oil from *Z. officinale* rhizome using MAHD method have been presented in Table 3. MAHD had smaller electrical consumption (0.11, 0.19, and 0.27 kWh for power level 180, 360, and 600 W) compared to HD (1.44 kWh), as well as, smaller CO_2 emission (0.09, 0.15, and 0.22 kg for MAHD at 180, 360, and 600 W, respectively, and 1.15 kg for HD). Using the MAHD method energy saving was from 84.75 to 91.15 % compared to the HD, while CO_2 emission was reduced for 81.19 to 92.28 %. Electrical consumption and CO_2 emission are related to the extraction time. Since the extraction time, the required energy, and CO_2 emission for MAHD are significantly reduced compared to HD, it is affirmed as a more environmentally friendly approach.

CONCLUSION

Yield of ginger rhizome essential oil improvement was achieved using MAHD extraction method, with no significant differences in the chemical composition comparing with conventional HD. About 80 % of the identified compounds belong to the group of non-oxygenated terpenes with α-zingiberene, camphene, β-phellandrene and 1,8-cineole as dominant compounds. Extraction time was significantly reduced in the case of MAHD (27-37 min compared to 144 min). MAHD has been shown as more environmentally friendly approach with electrical consumption 0.11-0.27 kWh compared to 1.44 kWh and CO_2 emission 0.09-0.22 kg compared to 1.14 kg. As hydrolat represent a by-product of HD process, additional aim of this study was also to investigate the chemical composition of obtained hydrolat so that its use can be indicated for different product of cosmetic and pharmaceutical industries. The dominant compound in hydrolats obtained by HD and MAHD at 180 W was 1,8-cineol, while in hydrolats obtained by MAHD at 360 and 600 W was α-zingiberene. Considering that the main component of ginger essential oil is α-zingiberene, and its content was the highest in essential oil and hydrolat obtained by MAHD at 600 W, it can be marked as the best of tested power level. Therefore, it can be concluded that MAHD is a good alternative for the extraction of essential oil from *Z. officinale* rhizome since it provides essential oils of similar quality to conventional HD while reducing the time of the process, electrical consumption, CO_2 emission, and increase the yields.

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