Comparative of Chemical Composition and Energy of *Zingiber officinale* Rosc. Essential Oil using Conventional and Microwave Hydro Diffusion-Gravity Methods

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Abstract

In this study, the optimal conditions of microwave hydro-diffusion and gravity (MHG) to extract essential oil from Zingiber officinale Rosc. (Ginger) were investigated. The MHG was compared with hydro-distillation (HD) in terms of extraction time, chemical composition, cost, and energy consumption. The results showed that the MHG optimized conditions occurred in 720 Watt of microwave power and 35 minutes of extraction time. Additionally, the comparison between MHG and HD methods revealed that the former spent shorter time (35 minutes) than the latter (180 minutes). The MHG method also spent less extraction cost and consumed less energy while provided more chemical composition (45 compounds for MHG and 4 compounds for HD).

Keywords: Green extraction, Hydro-diffusion, Solvent-free microwave extraction, Essential oil, Ginger

1. Introduction

Hydro-distillation method is the most commonly used for the extraction of essential oils from aromatic plants. However, the main problem of hydro-distillation is the extensive time spent for extraction which can cause a degradation of c thermally-sensitive compounds, and consequently decrease the efficiency of products [1]. High solvent consumption, high energy consumption, high waste and high cost [2] are also drawbacks of this method. These disadvantages have led to the need for better methods which save time and energy such as supercritical fluid extraction [3], subcritical water extraction [4] and microwave-assisted extraction [1]. Microwave-assisted extraction (MAE) is one of the improved methods that use microwave energy to extract essential oils resulting in less energy consumption, shorter extraction time, less water consumption and lower cost [1-2,5]. There are several methods based on microwave-assisted extraction such as microwave hydro-distillation (MAHD) [6], solvent-free microwave extraction (SFME) [7-10], green and solvent-free ultrasonic microwave-assisted extraction (UMAE) [11], microwave dry-diffusion and gravity (MDG) [12] and microwave hydro-diffusion and gravity (MHG) [13].

Microwave hydro-diffusion and gravity (MHG) is one of solvent-free microwave extraction techniques which was developed in 2008 by Chemat et al. [14]. This method extracts essential oils from fresh plant materials without adding any solvent or water. When receiving energy in the form of microwave energy, water and natural compounds in plant material evaporate and be condensed and dropped

by earth gravity [12] into a collector. The MHG method has a number of advantages such as simpler procedures, less energy consumption and higher purity of products [15]. In recent year, there have been a lot of researchers studying the extraction of aromatic plants using microwave hydro-diffusion and gravity method, for example, Vian et al. (2008) studied the extraction of essential oils from spearmint (*Mentha spicata* L.) and pennyroyal (*Mentha pulegium* L.) [14], Bourbia et al. (2009) studied the essential oils extracted from *Citrus* peels [16] and rosemary leaves [17], Benmoussa et al. (2018) studied the optimization of MHG extraction process of essential oil from Tunisian cumin (*Cuminum cyminum* L.) seeds [13] and Razzaghi et al.(2019) compared the MHG process with five several extraction processes of orange peel oil [18].

Ginger (*Zingiber officinale* Rosc.) belonging to the Zingiberaceae family is a common plant in Southern Asia [19]. Ginger rhizomes are of benefit to various applications such as culinary, spices, beverages, cosmetics and traditional medicine [19-21]. Rhizomes of ginger contain the highest yield of essential oil compared with other parts of it [22-23]. Chemical composition of ginger oil contains α - zingiberene, β - sesquiphellandrene, β – bisabolene, α – farnesene, camphene, cineol, geraniol, citral, etc. [19]. However, the amount of essential oil and the number of compounds obtained from ginger are varited depending on planting area, growth environment, harvest time, part of plant materials, fresh or dried plants and especially extraction method [20]. There are several methods for extracting essential oil from ginger, for example, hydro-distillation [24-26], steam distillation [27-29], supercritical carbon dioxide [3], subcritical water [4] and solvent extraction [20] and soon. In 2017, Racoti et al. [30] extracted ginger using microwave-assisted hydro-distillation (MAHD) method and solvent-free in situ microwave generated hydro-distillation method to study the conditions that give the best quality and quantity of products. Since is one of the most common plants grown in South-East Asia, there are economic pressure and interest in finding the most effective method to extract essential oil from gingers for the sake of market opportunity and higher income for ginger growers. For the above reasons, we are interested in studying the extraction of volatile oil from ginger.

The objective of this study was to optimize the conditions of microwave hydro-diffusion and gravity (MHG) using modify-microwave oven to MHG apparatus to extract essential oil from *Zingiber officinale* Rosc. (Ginger) rhizomes. Essential oils extracted from microwave hydro-diffusion and gravity and hydro-distillation were compared in terms of extraction time, yield, chemical compositions, cost, and energy consumption. The results of this study can be used as the reference to compare with those of other studies to identify the most suitable and effective method to extract ginger essential oil.

2. Materials and methods

2.1. Plant material

Fresh ginger rhizomes (approximate age of 12 months) were purchased from a market in Khon Kaen province, Thailand. The fresh rhizomes of ginger were washed, air - dried overnight and then sliced. For the MHG method, the rhizomes were sliced into 1-2 mm thick. For the HD method, the rhizomes were chopped into smaller pieces at $50 \pm 5 mm^3$ in order to increase surface area which consequently increases the efficiency of ginger extraction. The initial moisture content for sliced gingers and chopped gingers analyzed based on AOAC (2000) [20], is 85 – 87 %, respectively

2.2. Extraction methods

2.2.1 Microwave hydro-diffusion apparatus and procedure

Microwave hydro-diffusion and gravity (MHG) was performed in a modified commercial microwave oven (LG, 2.45GHz) as shown in Figure 1. It is a multimode microwave with a maximum power of 900 Watt. The sample reactor is made from Pyrex glass with a capacity of 3000 ml. Time and power are controlled by software.

In a typical MHG procedure performed at atmospheric pressure, a batch of 500-g fresh sliced ginger rhizomes was heated in a multimode microwave reactor without adding solvent or water. Essential oil and aromatic water (hydrosol) thus move naturally downwards by earth gravity on an Allihn condenser. The essential oil and hydrosol were collected and stored at 4 °C until analyzed.

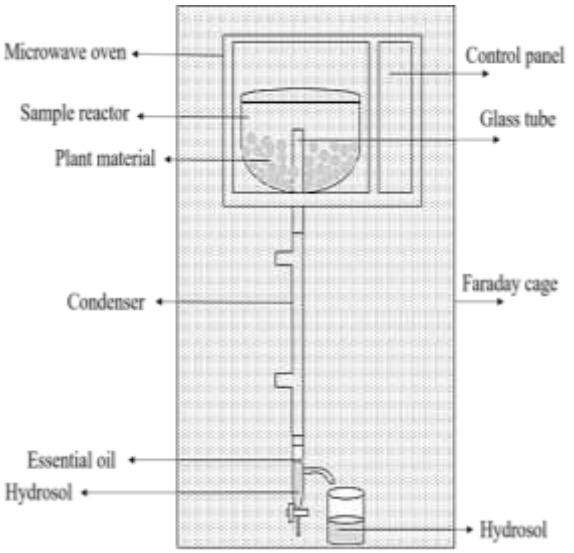

Figure 1 Schematic diagram of Microwave hydro-diffusion and gravity (MHG) process

2.2.2 Hydro-distillation apparatus and procedure

Fifty-gram of fresh chopped ginger rhizomes were subjected to hydro-distillation (HD) using a Clevenger – type apparatus. The HD was applied to chopped ginger rhizomes in 250 - ml distilled water for 3 h (or until no more essential oil was obtained). The essential oil and hydrosol were collected and stored at 4 °C until analyzed.

2.3. Gas chromatography – mass spectrometry

The composition of essential oils and hydrosols extracted by MHG and HD were determined by gas chromatography-mass spectrometry (GC-MS) (Bruker 450-GC gas chromatograph with a Bruker 320-MS mass spectrometer) fitted with Rtx-5MS fused silica capillary column (30 m x 0.25 mm x 0.25 µm film thickness). Helium was the carrier gas at a flow rate of 1 ml/min. The injection temperature was 280 °C. The oven temperature was adjusted from 60 to 250°C at increasing rate of 3°C/min and the total analysis was 64 min. For the analysis of essential oils extracted using MHG and HD methods, methanol was injected as a dilute in a split ratio of 1:50. On the other side, hydrosols were injected in the same ratio without dilute.

Chemical composition was identified by comparing of their mass spectra with those recorded in NIST (National Institute of Standard and Technology).

2.4. Energy

To calculate the electric consumption consumed in the extraction process, the formula below was used

Electric consumption (kWh)

= Power (kW) x Extraction time (h) (1)

3. Results and discussions

3.1 Preliminary of optimization of microwave power

The optimal conditions for the extraction of ginger essential oil under various microwave power of 540 W, 720 W and 900 W were examined. Figure 2 shows the extraction time as a function of the microwave power. The 900 W microwave power spent the shortest extraction time at 30 min. However, Filly et al. [7] reported that high microwave power can degrade chemical composition of essential oil and plant materials. Thus, when considering yield profiles as a function of time with different levels of microwave power as shown in Figure 2, it was found that the highest yield was produced at a 720 W microwave power. Although the 900 W microwave power spent shorter extraction time, lower yield was produced. The factor probably resulting in a decrease of yield on microwave power of 900 W that plant material burnt during the extraction time at high level of microwave power affects the amount of essential oil, smell and chemical composition [7,9]. Based on data from this study, microwave power of 720 W as an optimum power for the extraction of ginger essential oil spent 35 min.

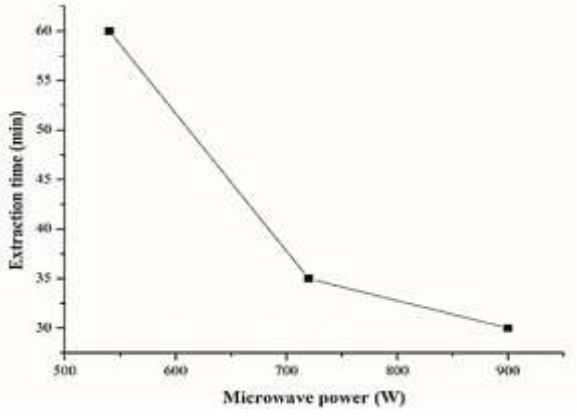

Figure 2 Microwave power as a function of extraction time with MHG

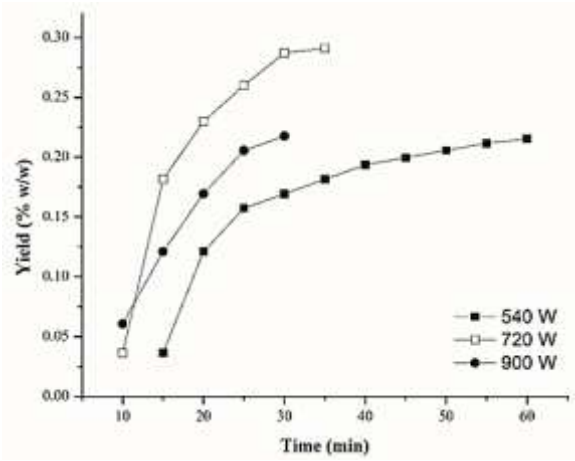

Figure 3 Yield profiles as a function of time for MHG extraction at different microwave power

3.2 Comparison of extraction kinetics and extraction yield

The comparison of kinetics of ginger essential oil extracted using MHG and HD is presented in Figure 4. The overall yields of the essential oils was obtained from ginger rhizomes by the MHG (720 W, 35 min) and the HD (180 min). The ultimate yields of essential oils obtained by the MHG and the HD method were 0.292% and 0.259%, respectively.

According to Figure 4, essential oil from HD method began to drip after 30 minutes while 95 % of that from MHG method was extracted during the range of time. As shown from the results of this study, MHG spent only 35 minutes and yield up to 1.12 times compared with HD which required 180 minutes for the extraction.

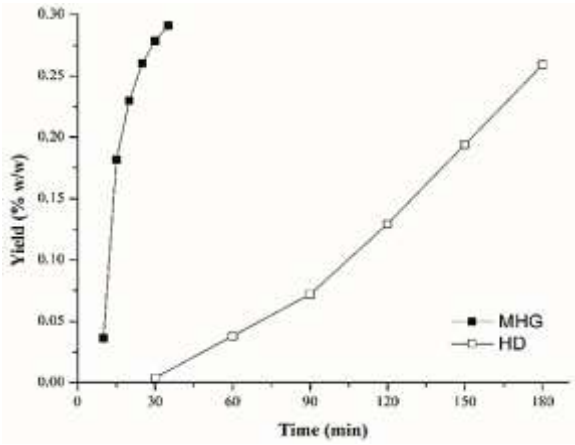

Figure 4 Extraction yield as a function of time for MHG and HD extraction of essential oil from *Zingiber officinale* Rosc rhizomes

3.3 Chemical composition of ginger essential oil and hydrosol

The chemical composition of the ginger essential oils and hydrosols obtained by the MHG under the optimum condition (720 W, 35 min), and by the HD (180 min) and the relative percentage values of the identified compounds in the essential oils and hydrosols were reported in Table 1. Ginger essential oils extracted by MHG and HD methods were significantly different in terms of their chemical composition. A total of 45 and 4 chemical compounds were identified in the GC-MS analysis of 100 % and 99.99 % of ginger essential oils, extracted from MHG and HD methods, respectively. The major compounds in the ginger essential oils extracted by MHG are geranial (30.45%), β-citral (17.49%), β-phellandrene (5.16%), eucalyptol (4.92%) and α-zingiberene (4.66%), respectively. The 4 components found from the HD method are geraniol, α-terpineol, borneol, and β-citral. The compounds found in the essential oils extracted from both methods are in accordance with those of Srinivasan et al. [19] and Racoti et al. [30]. However, the number and amount of components found in this research are inconsistent with both of the above studies due to various factors affecting the quantity of the compounds such as cultivated area, growth environment, harvest time and methods used to extract essential oils [7,30-31]. The reasons why only 4 compounds were found in ginger essential oils extracted from the HD method could be explained by Limpaphayom's research [20] that prolonged exposure high heat during the extraction can cause degradation of thermally sensitive compounds. Since ginger essential oil from HD extraction in this study exposed to high heat for a long period of time, only 4 compounds were found.

In the ginger rhizomes extraction, hydrosol is a by-product that occurs during the process. Hydrosols are a mixture of a quantity of essential oil and water-soluble which has previously been defined as waste from the process [32]. Nowadays, however, there are many researchers reconsidered the benefits of hydrosol in various plants. Therefore, in this research, we also studied the compounds in hydrosols in order to compare their chemical composition of ginger essential oil extracted from the MHG and HD methods. As shown in Table 1, 42 and 37 components were found in hydrosols extracted from MHG and HD methods, respectively. These findings correspond to those reported by De Matos et al. [32]

Table 1 Chemical composition of ginger essential oils and hydrosols obtained by MHG and HD

No.	RT[a] (min)	Compound[b]	Relative peak area[c] (%)			
			Essential oil		Hydrosol	
			HD	MHG	HD	MHG
1	5.85	2-Heptanol	-	0.37	0.17	0.32
2	7.03	α-Pinene	-	1.33	-	-
3	7.60	Camphene	-	4.52	-	-
4	8.71	β-Pinene	-	0.41	-	-
5	9.18	Sulcatone	-	0.68	0.13	0.75
6	9.35	β-Myrcene	-	1.90	-	-
7	9.49	Sulcatol	-	-	-	0.12
8	11.02	β-Phellandrene	-	5.16	1.68	4.73
9	11.11	Eucalyptol	-	4.92	-	-
10	13.75	Terpinolene	-	0.31	-	-
11	13.97	2-Nonanone	-	0.29	-	0.12
12	14.19	D-Verbenone	-	0.30	-	-
13	14.38	Linalool	-	1.25	0.48	1.31
14	14.44	2-Nonanol	-	0.68	-	-
15	16.85	Citronellal	-	0.29	-	0.15
16	17.45	Borneol	22.25	1.94	1.06	1.35
17	17.96	Terpene-4-ol	-	0.20	0.14	0.36
18	18.26	Limonene oxide	-	0.51	0.13	0.42
19	18.65	α-Terpineol	23.81	1.58	1.51	1.59
20	20.58	Citronellol	-	0.73	3.54	2.91
21	21.13	β-Citral	16.8	17.49	16.77	22.97
22	21.72	Geraniol	-	2.49	9.89	4.58
23	22.59	Geranial	37.13	30.45	50.01	40.11
24	23.39	2-Undecanone	-	0.79	-	-
25	27.59	β-Elemene	-	0.21	-	-
26	28.44	cis-1,1-diethoxy-3-hexene	-	0.34	-	-
27	29.94	Ethyl diethoxyacetata	-	0.36	-	-
28	30.12	Isoeugenol	-	-	0.21	0.25
29	31.24	β-Himachalene	-	-	0.18	0.23
30	31.27	Germacrene D	-	0.27	-	-
31	31.38	α-Curcumene	-	1.92	-	-
32	31.74	β-Eudesmene	-	0.29	-	-
33	31.93	α-Zingiberene	-	4.66	0.38	0.44
34	32.11	Isoeugenol methyl ether	-	-	0.26	0.24
35	32.44	α-Farnesene	-	4.09	-	-
36	32.87	Geraniol acetate	-	-	0.47	0.43
37	33.05	β-Sesquiphellandrene	-	2.79	0.85	0.92
38	33.56	trans-Edulan	-	0.20	0.28	0.41
39	34.03	Hedycaryol	-	0.62	2.23	2.3
40	34.58	Nerolidol	-	1.01	0.59	0.88
41	35.14	2,6-Dimethyl-1,6-heptadien-4-ol acetate	-	-	-	1.37
42	35.60	Sesquisabinene hydrate	-	0.31	0.65	0.77
43	36.58	α-Bisabolol	-	0.82	0.56	0.75
44	36.70	Epi-Eudesmol	-	0.26	0.16	0.27
45	36.83	Germacrene D-4-ol	-	-	0.30	0.26
46	36.98	Globulol	-	0.38	1.15	1.37
47	37.15	Epi-Globulol	-	0.50	1.04	0.78
48	37.34	Triquinacene, 1,4-bis(methoxy)	-	-	-	0.45
49	37.65	tau-Muurolol	-	-	0.12	0.22
50	37.85	α-Eudesmol	-	1.02	1.79	2.09
51	38.11	α-Cadinol	-	-	0.64	0.73

Table 1 *(continued)*

No.	RT[a] (min)	Compound[b]	Relative peak area[c] (%)			
			Essential oil		Hydrosol	
			HD	MHG	HD	MHG
52	38.16	Viridiflorol	-	0.30	0.41	0.47
53	38.41	ar-tumerone	-	0.22	0.16	0.74
54	38.56	Turmerone	-	0.31	0.14	0.25
55	39.28	Farnesol	-	0.53	1.13	0.88
56	39.58	6,10-Dodecadien-1-yn-3-ol,3,7,11-trimethyl	-	-	0.56	0.45
57	39.77	Curlone	-	-	0.08	0.18
58	41.18	Farnesal	-	-	0.16	0.11
Chemical component			4	45	37	42
Monoterpene hydrocarbon			-	13.32	1.68	4.7
Monoterpene oxygenated			99.99	61.87	83.74	75.85
Sesquiterpene hydrocarbon			-	15.05	2.78	2.91
Sesquiterpene oxygenated			-	4.56	7.1	8.96
Other			-	5.2	4.7	7.58
Total oxygenated compound			99.99	71.63	95.54	92.39
Total non-oxygenated compound			-	28.37	4.46	7.61
Total			99.99	100	100	100

HD: hydrodistillation method; MHG: microwave hydro-diffusion and gravity method

-: absence of compound

[a] Retention time listed in order to elution

[b] Essential oils and hydrosols compounds listed in order to elution

[c] Relative area percentage (peak area relative to the total peak area %)

which essential oil and hydrosol obtained from distillation have the same major components. The majority of compounds in hydrosols extracted by the MHG method are geranial (40.11%), β-citral (22.97%), β-phellandrene (4.73%), geraniol (4.58%) and citronellol (2.91%). The majority of compounds found in hydrosols extracted from HD method were geranial and β-citral. This is similar to the chemical compounds extracted from the MHG method and the compounds found in the following order are geraniol, citronellol, and hedycaryo, respectively. Additionally, it can be seen that the hydrosol extracted from the HD method contained more compounds than essential oil did from the same method. Because most substances tend to be soluble in water, extracted from HD method, ginger rhizome was directly exposed to boiling water causing the dissolution of compounds. Besides, some thermally-sensitive compounds could be degraded, resulting in the small number of compounds found in essential oil extracted from HD method.

3.4 Energy Consumption

Table 2 shows the comparison of MHG and HD extraction methods in term of energy consumption. MHG process required an extraction time of 35 min without adding water while the HD process required 180 min with 250 ml of water. This clearly points that the MHG method saves extraction time. When considering energy consumption of both extraction methods, 0.75 kWh and 0.42 kWh were used for HD and MHG methods, respectively. This can be concluded that the MHG method saves both time and energy resulting in less extraction cost. This corresponds to the research finding of Benmoussa et al.[13] which showed that the extraction of essential oil from Tunisian cumin seed using the MHG method consumed less energy than the HD method.

Table 2 Microwave hydro-diffusion and gravity compare with hydro-distillation

	HD	MHG
Power (W)	250	720
Power per load (W/kg)	5	1.4
Extraction time (min)	180	35
Electric consumption (kWh)	0.75	0.42
Electric consumption per load (kWh/kg)	15	0.84
Water adding (ml)	250	0
Yield (%w/w)	0.259	0.292

4. Conclusions

In this research study, microwave hydro-diffusion and gravity (MHG) and hydro-distillation (HD) techniques were used to extract rhizomes essential oil of Zingiber officinale Rosc. The results of this study confirm that microwave hydro-diffusion and gravity seemed beneficial in terms of extraction time, chemical composition, and energy cost. In other words, the optimum power of MHG required to extract essential oil from 500 g of Zingiber officinale Rosc. is 720 W in 35 min. with a yield of 0.292%, Under these conditions 45 chemical compositions of essential oil were obtained. Moreover, the MHG procedure for essential oil extraction is simpler than that of HD method because it is solvent-free and requires no extra step of chopping rhizomes after slicing. Lastly, the MHG method, is suitable for the extraction of essential oil from various aromatic plants because it is fast, economical and efficient.

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