MICROWAVE HYDRODIFFUSION AND GRAVITY, A GREEN METHOD FOR THE ESSENTIAL OIL EXTRACTION FROM GINGER – ENERGY CONSIDERATIONS

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In this work was studied the extraction of ginger essential oil (EO) by microwave hydrodiffusion and gravity (MHG) method. The influence of specific absorption rate (SAR) and the addition of saturated steam into the system on the extraction efficiency were followed. A comparative extraction by microwave hydrodistillation (MHD) was performed. The best results were achieved for a moderate SAR value and by addition of saturated steam into the system. The extraction time and the specific microwave energy for MHG were approximately 5 times, respectively 3 times lower than MHD.

Keywords: microwave hydrodiffusion and gravity, specific absorption rate, ginger, essential oil

1. Introduction

Ginger (*Zingiber officinale*) which belongs to *Zingiberaceae* family is a slender perennial herb with fibrous roots, cultivated on a large scale worldwide including India, North East Asia, Australia, China, Jamaica and Japan [1,2].

Ginger rhizome shows many beneficial health properties that are valuable in medicine. Also, it is one of the most used spices of ginger family and common condiment for foods and beverages due to its distinctive pungency and piquant flavor [3]. Ginger rhizome contains carbohydrates, protein, crude fiber, fatty oil, polyphenols, and essential oil. The compounds which give its characteristic flavor are gingerols, shogaols, zingerone, and essential oils that comprise up to 3% of

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ginger. The volatile oil contains mainly sesquiterpenoids, with α-zingiberene as the main component, monoterpenoids such as β- phellandrene, camphene, cineol, etc., diterpenes and ginger glycolipids. These bioactive compounds present antioxidative, anti-inflammatory, anti-carcinogenic, antiseptic, anti-hyperglycemic, anti-lipidemic, and immune-modulatory properties [4,5,6,7].

The common classical methods used for the essential oil extraction from plant materials are hydrodistillation, steamdistillation, and solvent extraction. Due to several shortcomings of these methods, such as large amount of solvent consumption with the possibility of leaving toxic solvent residue in essential oils, longer extraction time which may degrade the oil quality, and high energy consumption, many researches were conducted for alternative techniques of the essential oil extraction [8,9,10,11,12]. These new approaches include microwave assisted extraction (MAE), ultrasound assisted extraction (UAE), pressurized solvent extraction, supercritical fluid extraction, solvent-free extraction, and microwave hydrodiffusion and gravity extraction (MHG) [13,14,15,16].

Nowadays, the microwave technique for essential oils and natural products extraction has brought much attention within scientist community. The advantages of MAE consist of high yield and selectivity, short extraction times, more effective heating, and low energy consumption [17,18]. The MHG method was successfully tested for the essential oil extraction from plant materials [19,20,21]. The principle of this approach is based on the microwave heating combined with earth gravity. During MHG the plant cell is swollen by heating the constituent water. Further the cell's membrane is disrupted, and the compounds of interest diffuse outside the plant tissue [22,23].

This work describes the extraction of the essential oil from ginger rhizomes by MHG method using an innovative experimental installation provided with a stirring system and which allows the addition of saturated steam. The purpose of this study was to follow the extraction efficiencies considering the energy consumption.

2. Material and methods

2.1. Plant Material

The fresh ginger rhizomes used in this research were purchased from local market in Bucharest, Romania. The material was kept at 4 ºC until extraction. The experiments were performed using as feedstock shredded ginger rhizomes and pressed pulp resulted from mechanical pressing of ginger rhizomes. The initial content of water of fresh ginger rhizome was approximatively 90%. Applying a mechanical pressing to 100 g ginger rhizomes results 75 g pressed pulp which has an initial content of water of approximatively 70%.

2.2. Microwave extraction equipment

An innovative experimental installation (IEI) was specially designed and built for fresh plant materials treatment by MHG (Fig. 1).

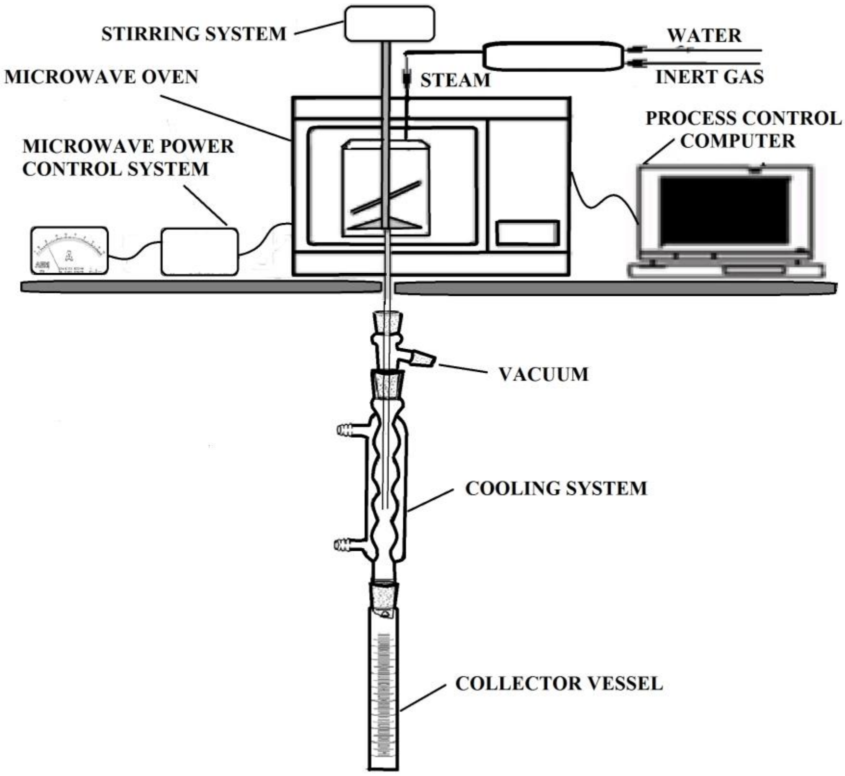


Fig. 1. The innovative experimental installation based on MHG method

The innovation of IEI, compared with classical MHG equipment cited in the literature, consists of a stirring system.

The equipment is provided with a treatment reactor with a capacity of 100-500 g. IEI allows continuous setting of microwave power between 0-700 W, continuous plant stirring (to achieve a uniform microwave irradiation and to avoid local degradation and overheating), temperature monitoring with an infrared device, and introducing saturated steam or inert gases into the system. The vapors resulted after treatment, condense and the EO is separated by gravity. The experiments can be performed at normal or low pressure.

2.3. Essential oil extraction procedure

The extraction of the EO was carried out by two methods: MHD and MHG. MHD was used as reference method to compare the amount of the EO resulted after the extraction by MHG. Due to low amounts of EO, its quantification from the obtained extraction mixture (water and EO) was performed by extraction with hexane in three cycles using a 1/3 (v/v) ratio of hexane to extraction mixture. The resulted mixture (hexane and EO) was kept at a temperature of 4 ºC until GC/MS analysis.

Microwave hydrodiffusion and gravity

The extraction of the EO from ginger by MHG was performed using the innovative experimental installation presented in Fig. 1.

For each experiment 200 g of plant material (shredded ginger rhizomes or pressed pulp resulted from mechanical pressing of ginger rhizomes) were submitted to MHG. The extractions were carried out at constant power (460 or 680 W) or by changing the power during the process (from 205 to 680 W) for 13 to 25 min. The maximum temperature was approximatively 106 – 108ºC for all experiments. In order to improve the EO training, saturated steam was introduced into the system. The cooling agent temperature was 5°C for all experiments. During the extractions temperature, microwave power and time were recorded using an operating console (software LabView).

Microwave hydrodistillation

The extraction of the EO by MHD was carried out using a multimode microwave oven (Plazmatronika, Poland). Fresh plant materials (200 g of shredded ginger rhizomes and 100 g of pressed pulp) were submitted to MHD using a Neo Clevenger type apparatus fitted with a 10 mL graduated tube. The EO was extracted for 105 min. For shredded ginger rhizomes a 2/1 ratio of water to plant was used and for pressed pulp a 3/1 ratio respectively.

2.4. GC-MS analysis of ginger EO

The quantitative analysis of the EO was performed using a HP 6890 gas chromatograph equipped with flame ionization detector (FID). The separation was carried out on a DB-1 (J&W Scientific) 50m×0.250mm×0.5µm capillary column. The oven was set to heat the column from 50ºC to 250ºC with a gradient of 10ºC/min. Helium was used as carrier gas through the column (1 mL/min flow rate). n-Nonane was used as internal standard for the determination of EO concentrations.

2.5. SEM analysis

Scanning electron microscopy images (SEM) were obtained using a Quanta Inspect F microscope from FEI Company with field emission gun (FEG) and a 1.2-nm resolution, equipped with an energy-dispersive X-ray spectrometer (EDX) with an energy resolution of 133 eV (Mn Ka).

2.6. Determination of specific microwave energy and extraction efficiency

In order to highlight the advantage of the MHG process compared with MHD, several parameters were calculated (specific microwave energy supplied by the magnetron, specific microwave energy absorbed by the sample, total efficiency of EO obtained from pressed pulp ginger, and separation efficiency of the EO in distillate).

The determinations on the absorbed microwave power (established by water calorimetry) depending on the actual microwave power supplied by the magnetron, show a significant decrease of the absorbed microwave power when the mass sample is reduced below 200 g (see Fig. 2). This data are consistent with literature [24,25].

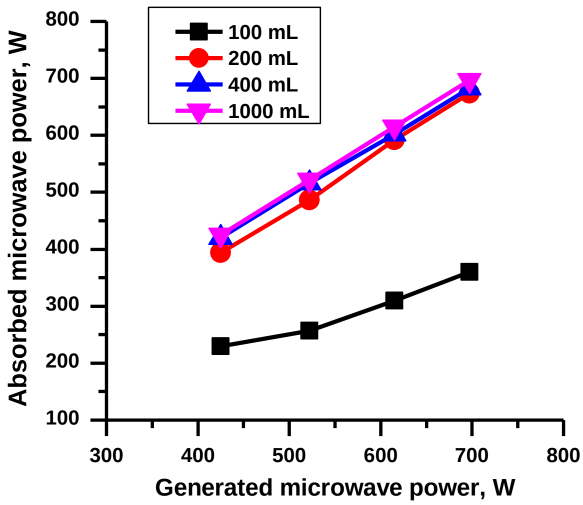


Fig. 2. Microwave power absorbed by different volumes of water at different level of generated microwave power

The specific microwave energy supplied by the magnetron and the specific microwave energy absorbed by the sample were calculated using the following equations:

$$E_{magn\_sp} = \frac{E_{magn}}{V_{EO\_d}} \quad (1)$$

$$E_{ab\_sp} = \frac{E_{ab}}{V_{EO\_d}} \quad (2)$$

where: E_{magn_sp} – specific microwave energy supplied by the magnetron related to the EO obtained from 100 g feedstock (kJ/mL); E_{magn} – microwave energy supplied by the magnetron (kJ); V_{EO_d} – EO volume in distillate obtained from 100 g feedstock (mL/100g); E_{ab_sp} – specific microwave energy absorbed by the sample related to the EO obtained from 100 g feedstock (kJ/mL); E_{ab} – microwave energy absorbed by the sample (kJ).

The total efficiency of the EO obtained from pressed pulp ginger and separation efficiency of the EO in distillate were calculated using the following equations:

$$Ef_1 = \frac{EO_{MHG\_d} + EO_{MHG\_p}}{EO_{MHD}} \times 100 \qquad (3)$$

$$Ef_2 = \frac{EO_{MHG\_d}}{EO_{MHD\_d} + EO_{MHG\_p}} \times 100 \qquad (4)$$

where: Ef_1 – total extraction efficiency of the EO obtained from pressed pulp ginger (%); EO_{MHG_d} – EO volume in distillate obtained by MHG (mL), EO_{MHG_p} – EO volume remained in the ginger treated pulp (mL), EO_{MHD} – EO volume obtained by MHD method; Ef_2 – separation efficiency of the EO in distillate (%).

3. Result and discussion

3.1. Extraction of the EO from ginger rhizomes by MHD

The total amount of the EO found in shredded ginger rhizomes or pressed pulp was determined by MHD extraction (see Table 1). This approach was used as reference method to compare the amount of the EO resulted after MHG extraction. Thus, the extraction efficiencies for the total amount of the EO from shredded ginger rhizomes or pressed pulp was assumed to be 100%.

Table 1

Experimental conditions and results for MHD extraction of EO from shredded and pressed pulp ginger

Exp	Ginger type	P* [W]	SAR** [W/kg]	E_{magn_sp} [kJ/mL EO]	E_{ab_sp} [kJ/mL EO]	V_{EO_d} [mL/100g]
A.	Pressed pulp	380	0.8-0.5 x10^3	2284	2284	0.599
B.	Shredded ginger	380	0.5-0.3 x10^3	6080	6080	0.225

*P – supplied microwave power during the extraction; **SAR – specific absorption rate related to feedstock.

3.2. Extraction of the EO from ginger rhizomes by MHG

In our study the influence of SAR and the addition of saturated steam into the system on the extraction efficiency of ginger EO by MHG were followed. The experimental conditions and results are shown in Table 2. The evolution of SAR values are shown in Fig. 3.

Table 2

Experimental conditions and results for MHG extraction of EO from shredded and pressed pulp ginger

Exp	Ginger type	P [W]	SAR [W/kg]	E_{magn_sp} [kJ/mL EO]	E_{ab_sp} [kJ/mL EO]	V_{EO_d}*** [mL/100g]	V_{EO_p} [mL/100g]
1.	Shredded ginger	678	3.41-5.5 x10^3	2487.5	1216.7	0.21	0.054
2.	Shredded ginger	678-366	3.46-3.94 x10^3	2421.3	1059.9	0.191	0.103
3.	Pressed pulp	460	2.3-3.5 x10^3	1268	923.1	0.447	0.283
4.	Pressed pulp	432-205	2.16-1.45 x10^3	650.4	494.2	0.64	0.394
5.	Pressed pulp	615-423	3.07-2.55 x10^3	1370	904.1	0.376	0.521
6.	Pressed pulp	460	2.3-3.2 x10^3	960.3	759.9	0.619	0.417
7.	Pressed pulp	435-209	2.34-3.29 x10^3	699.5	533.6	0.648	0.359
8.	Pressed pulp	615-441	3.07-3.42 x10^3	1064.5	826.4	0.504	0.291

***V_{EO_p} – EO volum remained in the treated ginger related to 100 g feedstock.

The change of magnetron power during the extraction process is determined by a dramatic decrease of the mass sample (from 200 g to approximately 60 g for pressed pulp). This decrease in mass sample causes a significant raise of SAR whether the microwave power is maintained constant (see Fig. 3 exp. 1). Analyzing these data reveal that the experiments performed at variable SAR with limited values lead to better results in terms of the efficiency of EO extraction (see Fig. 4).

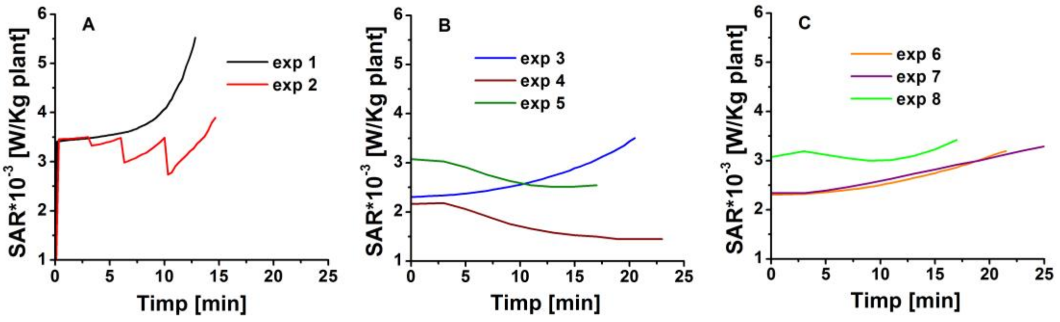


Fig. 3. SAR evolution during MHG (A – experiments with shredded ginger; B – experiments with pressed pulp without saturated steam; C – experiments with pressed pulp and addition of saturated steam into the system)

The total efficiency of the EO obtained from pressed pulp ginger and separation efficiency of the EO in distillate determined for all the experiments are shown in Fig. 4. For the best extraction conditions (exp. 4 and 7), the energy consumption is approximately 3 times lower than the classical extraction by MHD or than the experiments performed by MHG at constant power (see Fig. 4).

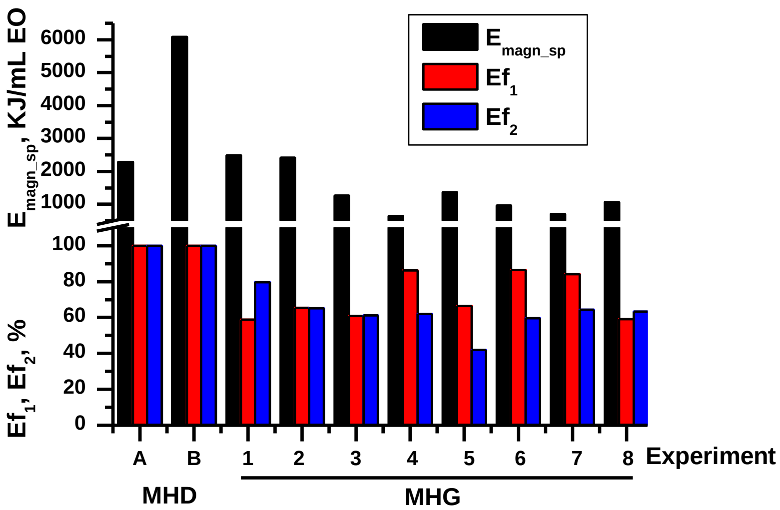
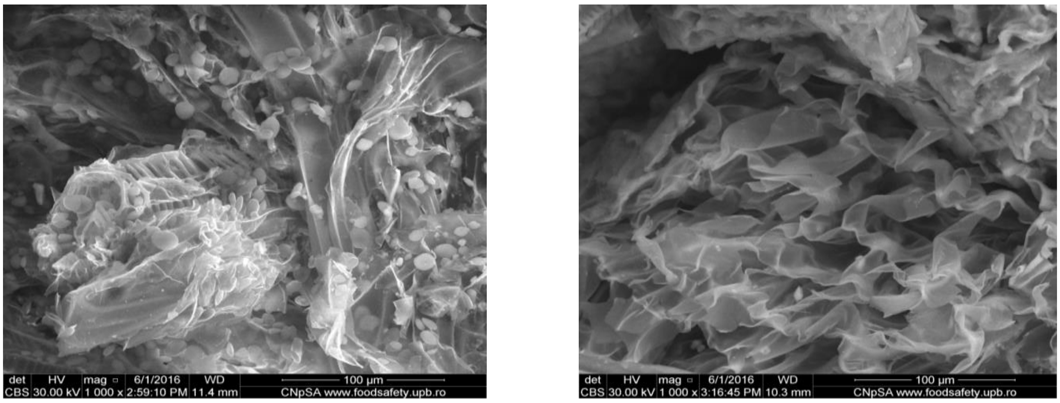


Fig. 4. Extraction efficiencies and specific microwave energy for MHG vs. MHD

3.3. SEM analysis

Fig. 5 shows the SEM images of untreated ginger pulp and pulp after MHG treatment. The essential oil is biosynthesized in specialized cells, such as glandular trichomes, osmophores, or ducts and cavities [26]. As shown in Fig. 5A the essential oil can be found in those small spherical vacuoles contained by the glandular trichome cells. After MHG treatment the vacuoles are no longer presented (see Fig. 5B).



A B

Fig. 5. SEM images of ginger pulp (A – untreated pulp, B – after MHG treatment)

4. Conclusions

MHG is an alternative technique for the extraction of EO which combines the microwave heating with earth gravity. For this extraction method, the addition of a solvent is not required.

The short residence time of feedstock into the extraction vessel represents an important advantage of this process. The compounds of interest are collected by gravity in liquid form, thus a long-time heating of feedstock to train the EO with water vapors is not necessary. The microwave applicator is a multimode type. Therefore, in order to achieve a uniform heating of plant material an efficient stirring system is required. Another important particularity of MHG process is the microwave specific absorption rate (SAR). Due to the significantly decrease of the mass sample during the process, the SAR value increases and consequently the EO yield is lower (for a constant microwave power).

The best results for MHG extraction of ginger EO were achieved by controlling the magnetron power during the process (to avoid excessive SAR value) and introducing saturated steam into the system. The extraction efficiencies were good enough compared with classical MHD method. However, considering the shorter extraction time (approximately 5 times lower than MHD) and the lower specific microwave energy (approximately 3 times lower than MHD) the MHG is a more energetic efficient extraction method.

Ginger contains a small amount of essential oil compared with other aromatic plants and its extraction can be difficult. Thus, further research in order to enhance the yield by MHG method is necessary.

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