ON THE EFFECT OF THE RATIO OF THE DISTILER VOLUME AND THAT OF THE MICROWAVE CAVITY ON THE EXTRACTION OF Cymbopogon nardus DRIED LEAVES BY MICROWAVE HYDRODISTILLATION

by Zuhdi Ma`sum .

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ABSTRACT

The production of essential oils as fragrances and aroma ingredients has been evolving towards an industrial scale for decades. One of the essential oils used in industry is a produce from citronella grass (Cymbopogon nardus). A study of a step up in capacity of Cymbopogon nardus microwave radiation treatment is undertaken. This research compares the effect of different parameters such as the microwave power and the ratio of distiler volume and that of the microwave cavity (D/C). With the aim to determine the relationship between the parameters, these research is performed using a microwave power of 700 W, 560 W, 430 W at D/C values of 0.08, 0.04, and 0.02 in case of a feed to solvent ratio (F/S) of 24 %. The results of these experiments indicate that the usage of microwave power and the increase of D/C ratio lead to an increase of the oil yields by up to 34 %, 25 %, and 34 %. The chemical composition of the citronella oil is analyzed by GC-MS. It is found containing mainly citronella, geraniol and citronellol of 40.54 %, 17.39 %, and 15.88 %, respectively. The results show that the increase of D/C ratio and the microwave power increase essentially the extraction yield. They also have significant implications for the continuing use of microwave radiation in extraction and could be compromised to scale up the capacity.

Keywords: microwave hydrodistillation, distiller to cavity (D/C), microwave power (W), step up capacity, Cymbopogon nardus.

INTRODUCTION

The essential oils of the grasses of genus of *Cymbopogon* have an industrial profile; they are used in beverages, foodstuffs, fragrances, household products, personal care products, pharmaceuticals. One of the most important among the 180 species of this genus refers to *Cymbopogon nardus*. This plant belongs to the family *Poaceae* from the *Liliopsida* class with a height of up to and above 1 m, with narrow and along leaves that are characterized by the presence of silica thorns aligned on the leaf edges. The leaves bear glandular hairs, usually each with a basal cell that is wider than the distal one. The extract of this plant contains essential oils, namely citronella oil or java citronella [1].

At present, various innovations referring to an alternative energy must be developed to replace conventional energy use which is found wasteful and ineffective. Some alternatives are advanced. They are connected with sources "not commonly" used as energy one. This is for example the radio frequency which is generally used in communications but currently finds an application in processes of sintering, drying, melting, defrosting, and extraction [2]. This uncommon energy is often called '*green energy*'. The term refers to a process leading to low pollution [3].

Microwave hydrodistillation as one of the many processes using green energy is among the major techniques used for extracting compounds from aromatic plants. Golmakani [4] reports some recent studies focused on the successful utilization of a microwave oven for the extraction of active components of a plant origin. The main advantage of the microwave hydrodistillation refers to the minimized energy use and decreased extraction time [5 - 7]. It is not only a new method but also has a potential to be developed on a larger scale [2, 8 - 13]. Microwave hydrodistillation has many advantages but it requires an increased quantity of the materials extracted which is a constraint.

Some researchers claim that the dielectric heating rates depend on the size, the geometry, the position, and the material during the extraction [14 - 16]. It is shown that the spherical geometry provides an equal temperature distribution on all surfaces. This study is intended to observe only the heating distribution in apparatuses of a various geometry and size. A research on the absorption of microwave energy by mineral solutions is also carried out and shows the effect of the sample volume [17]. The results obtained show that the absobed microwave energy increases with increase of the sample volume. Although this study is widely publicized, however, a research on increasing the ratio of the distiler volume and that of the microwave cavity (DC) has never been published.

The purpose of this study is to investigate how (D/C) ratio influences the yields and the extraction time of the aromatic plants extraction. Various D/C values are used to extract *Cymbopogon nardus* dried leaves using the microwave hydrodistillation method. They refer to 0.02, 0.04, and 0,08, while the feed-to-solvent (F/S) ratio is 0.24 (referring to the full capacity of the distiller).

EXPERIMENTAL

Microwave Apparatus

The domestic microwave (Fig. 1(a)) working at the frequency of 2.45 GHz, an wavelength of 12.24 cm and energy between 1.24x10^{-6} eV and 1.24x10^{-3} eV acted as an nonionizing radiation of no effect on the molecular structure. This energy was only absorbed by the water which had a tangent loss (tan δ) value of 9,889 and rose to a molecular rotation during the radiation. The molecular rotation occured at a very high speed. Through an ionic motion and a dipole rotation it could significantly shorten the heating process [18 - 21].

Dimention of the microwave cavity and the volume of the distiller

The microwave cavity was a rectangular closed metal box of dimensions of 330 mm x 360 mm x 210mm. The extraction chamber used a round distiller of a volume of 500 mL, 1000 mL and 2000 mL (Fig. 1(b)). The distance from the center to the wall of the distiller was 4.9 cm , 6.2 cm and 7.8 cm , respectively.

Microwave power

The microwave power input was equal to 1000W at a frequency at 2.45 GHz. The domestic microwave equipment converted this input electricity power to 70 % of microwave power, i.e. 700 W. The latter was the

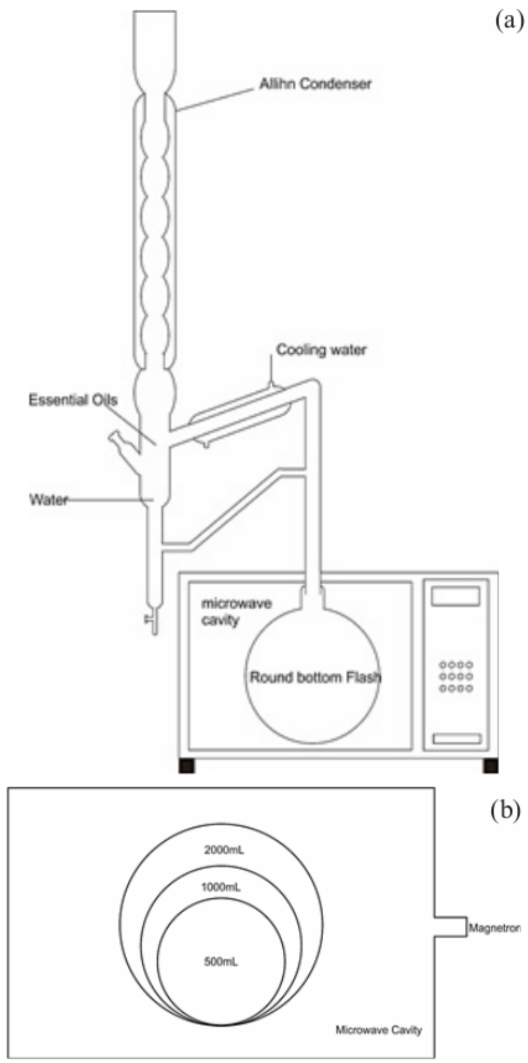

Fig. 1. (a) A microwave hydrodistillation apparatus; (b) a schematic presentation of the different volumes position inside the microwave cavity.

highest (100 %) used. The lower values used referred to 560 W and 420 W.

Microwave hydrodistillation

The extraction work carried out consisted of the following steps. The plant material was packed in the distiller and then water was added in a sufficient amount. The mixture obtained was brought to the center of the microwave cavity (Fig. 1(b)) [17]. The samples extracted strung together with Clevenger apparatus [23]. The preheating was measured from the moment the microwave was turned on till that of the evaporation beginning. The steam which came out from the extraction chamber was condensed by a condenser apparatus and transported to a separator, where the oil separated automatically from the water [24]. The yield was followed every 10 min after preheating. The process continued until reaching the maximum extraction time of 150 min.

Analysis

The microwave loading capacity was defined by the ratio of the volume of the distiller to that of the microwave cavity (D/C), i.e.

$$\text{Ratio of distiller to microwave cavity } \left(\frac{D}{C}\right) = $$

$$= \frac{\text{Volume of distiller}}{\text{Volume of microwave cavity}}$$

The distiller loading capacity was defined by the ratio of the weight of samples and that of the solvent, i.e.

$$\text{Ratio of feed to solvent } (F/S) = \frac{\text{Weight of samples}}{\text{Volume of solvent}}$$

The water in the extraction yields was removed using anhydrous sodium sulfate and cooled at 4°C. Yield percentage was determined by using the following equation:

$$\text{Yield } (\%) = \frac{\text{Weight of essential oil}}{\text{Weight of samples}} \cdot 100\ \%$$

The moisture content of the sample was measured using a moisture meter probe in the range of 2 % - 60 % with an accuracy of 1 %. Citronella oils composition was determined by gas chromatography coupled to mass spectrometry (GC–MS) analysis carried out on Agilent 6890N Network GC System, Agilent J&W Capillary.

Sample preparations

The plant of *Cymbopogon nardus* (citronella) collected from Pacet, Mojokerto East Java at an altitude of about 400 m - 600 m above the sea level was used. The plant grew naturally and harvested after growth of eight months. The harvesting was done by cutting the leaves and leaving the stump at the base. Leaves were separated from the plants and dried in an open space, avoiding a direct sun light, for ten days [22]. The dried leaves having a moisture content of 6,5 % were cut into pieces of 5 mm length.The drying process was carried out for a long time to ensure that the water content in the material did not significantly affect the absorption of solvents under electromagnetic conditions.

RESULTS AND DISCUSSION

Effect of the microwave power on the preheating

In this study, the preheating has a significant effect on the yield and the extraction time, especially at the beginning of the process. Preheating is the process through which the materials and the solvents in a distiller are irradiated at the initial temperature until solvent evaporation starts. The initial temperature value is equal to 27°C, while that of evaporation amounts to 115°C. Table 1 presents the duration of the preheating in case of different distiller volumes and shows that the higher the microwave power used, the shorter the heating time is [25].

Tabel 1. Comparison of the effects of the ratio of the distiller volume and that of the microwave cavity (D/C) and the microwave power on the heating time required.

Volume distiller to volume of microwave cavity (D/C)	MW Power(W)	Time (min)
0.08	700	4
0.08	560	5
0.08	420	8
0.04	700	3.5
0.04	560	4
0.04	420	7
0.02	700	2
0.02	560	3,5
0.02	420	4

The microwave used has a power of 700W which means that it has 60 s per min for a maximum radiation or an uninterrupted radiation. The lower microwave power of this device refers to 560 W and 420 W, which means it has 2x24 s and 3x18 s per min. So they have an interrupted radiation of 2x24 s per min and 2x18 s per min, respectively. When the radiation is interrupted, there is cooling time of 2x6 s and 2x12 s, respectively. This results in boiling temperature decrease by 17°C and 20°C, correspondingly. The reheating that follows the cooling time lag is slower because of weakened molecular movements due to increased viscosity of the solvent [5]. This circumstance explains the difference in the heating time.

Effect of D/C ratio on the preheating

The dependence of the heating time on D/C ratio (Fig. 2) obtained experimentally shows that the smaller ratio provides shorter heating times [26 - 28]. The heating time difference arises because of the limited penetration depth of the microwaves while the volume increases. So that for a small volume of the distiller, the heating occurs simulateneously at the surface of the wall and in the bulk of the distiller. By increasing the distiller volume, the heating in the bulk changes [16].

Effect of D/C ratio on the extraction yield

As explained earlier the preheating has a significant effect on the yield and the time of extraction. The graph of Fig. 3 shows that the amount of the extracted oil increases sharply within the initial 10 min and then decreases [29]. The exception observed in case a load ratio of 0.08 will be explained later. The yield refers to the accumulated oil not yet evaporated prior to reaching the evaporation temperature. A process of heating the plants surfaces at and near the inner walls of the distiller occurs [16] at the beginning of the radiation. It ruptures the plant walls which in fact results in the release of oil from the plant matrix [30].

The diffusing oil cannot immediately evaporate because the temperature has not reached that of the boiling point. The oil is evaporated when the latter is reached. However, at a load ratio of 0.08, the increase occurs in two stages: within 10 min and 20 min. In this case, the increase of the load ratio causes an expansion of the distiller surface and provides even heating at the surface of the volume [17]. Subsequently, a better heat distribution throughout the matrix increases the extraction yield. Expanding the geometric surface might alternatively resolve the problem of the limited microwave penetration depth.

The experimental results obtained are described as follows. At the loading capacity ratio of 0.02, the yield increases during first 10 min but decreases during the subsequent 20 min to 50 min. (Fig. 3(c)). During that time, the extraction proceeding at the lowest microwave power reaches the lowest yield, while the highest yield is obtained at microwave power of 560 W. At the end of the extraction, the measurement of the total yield shows that the lowest value amounts to 0.86 % at 420 W, while the highest one reaches 1.09 % in case of 560 W (Table 5). The extraction times are close (Table 2). The same pattern is observed at a loading capacity ratio of 0.04 (Fig. 3(b)). The yield generated at this ratio is not much different even though the usage of microwave power of

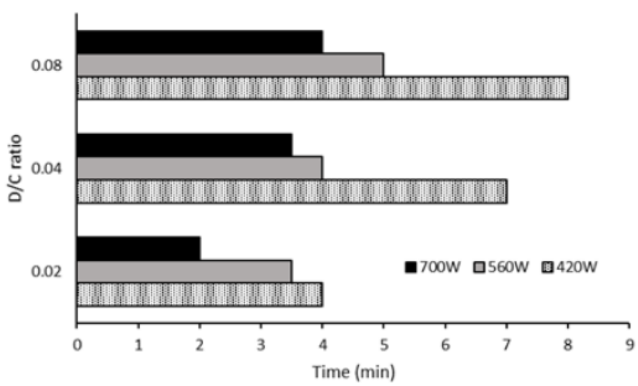

Fig. 2. D/C ratio vs the heating time at variuos power values.

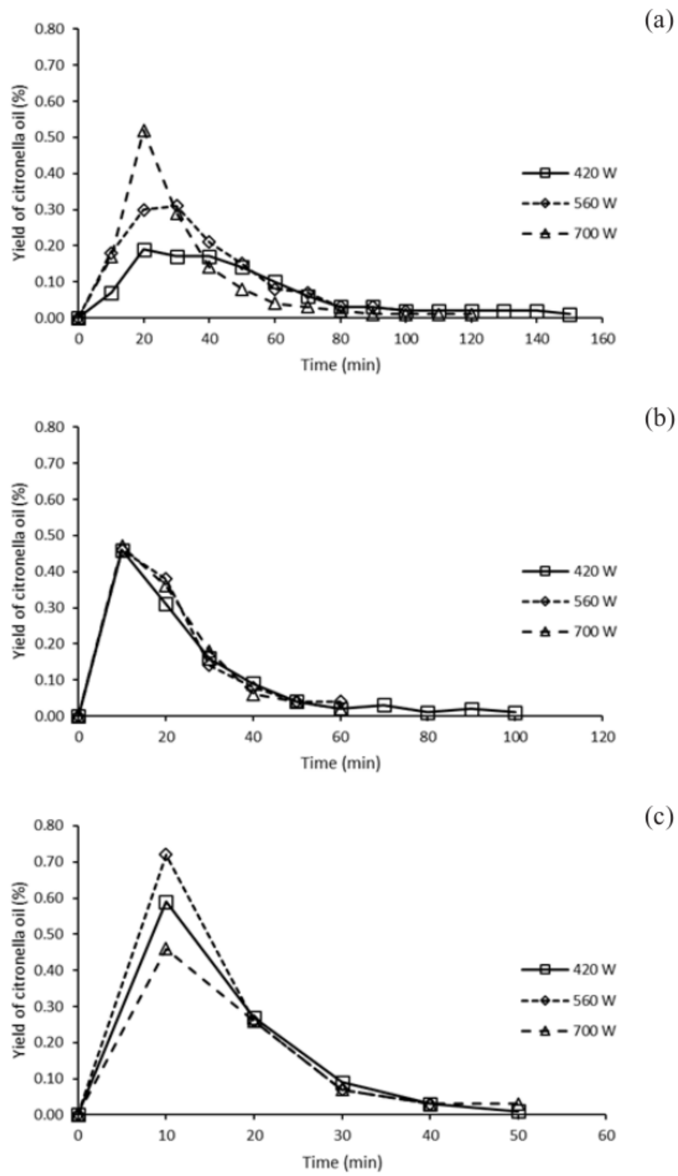

Fig. 3. A dependence of the extraction yield within 10 min interval at a material to a solvent ratio of 24 % and microwave power of 420 W, 560 W, 700 W for the ratio of the distiller volume and that of the microwave cavity of: (a) 0.08; (b) 0.04; (c) 0.02.

420W results in longer extraction time. The comparison of the total yields shows values of 1.12 %, 1.14 % and 1.15 % (Table 5) with extraction time of 60 min, 60 min, and 100 min corresponding to power of 700 W, 560 W, and 420 W (Table 3). The highest yield is obtained at a ratio of 0.08 with a significant increase within the intial 10 min and 20 min followed by a decrease to 30 min and 150 min (Fig. 3(a)). During the initial 10 min to 20 min, the microwave power of 420 W provides the lowest yield compared to that in case of 700 W. The total percentage yield at 420 W is also lowest (0.86 %) when compared to that obaserved in case of 560 W. The highest total

Table 2. A comparison of the yield obtained at a ratio of the distiller volume and that of the microwave cavity of 0.02.

Time, min	700W		560W		420W	
	Yield, %	accumulation yield, %	Yield, %	accumulation yield, %	Yield, %	accumulation yield,%
0	0.00	0.00	0.00	0.00	0.00	0.00
10	0.46	0.46	0.72	0.72	0.59	0.59
20	0.26	0.73	0.26	0.98	0.27	0.86
30	0.07	0.80	0.07	1.06	0.09	0.95
40	0.03	0.83	0.03	1.09	0.03	0.98
50	0.03	0.86	-	-	0.01	0.99

Table 3. A comparison of the yield obtained at a ratio of the distiller volume and that of the microwave cavity of 0.04.

Time, min	700W		560W		420W	
	Yield, %	accumulation yield, %	Yield, %	accumulation yield, %	Yield, %	accumulation yield,%
0	0.00	0.00	0.00	0.00	0.00	0.00
10	0.47	0.47	0.46	0.46	0.46	0.46
20	0.36	0.83	0.38	0.84	0.31	0.78
30	0.18	1.01	0.14	0.98	0.16	0.94
40	0.06	1.07	0.08	1.06	0.09	1.03
50	0.04	1.09	0.04	1.10	0.04	1.07
60	0.02	1.11	0.04	1.14	0.02	1.08
70	-	-	-	-	0.03	1.11
80	-	-	-	-	0.01	1.12
90	-	-	-	-	0.02	1.14
100	-	-	-	-	0.01	1.15

Table 4. A comparison of the yield obtained at a ratio of the distiller volume and that of the microwave cavity of 0.08.

Time, min	700W		560W		420W	
	Yield, %	accumulation yield,%	Yield, %	accumulation yield,%	Yield, %	accumulation yield, %
0	0.00	0.00	0.00	0.00	0.00	0.00
10	0.17	0.17	0.18	0.18	0.07	0.07
20	0.52	0.69	0.30	0.47	0.19	0.26
30	0.29	0.98	0.31	0.78	0.17	0.43
40	0.14	1.13	0.21	0.99	0.17	0.60
50	0.08	1.21	0.15	1.14	0.14	0.74
60	0.04	1.25	0.08	1.21	0.10	0.85
70	0.03	1.28	0.07	1.28	0.06	0.91
80	0.02	1.30	0.03	1.32	0.03	0.94
90	0.01	1.31	0.03	1.34	0.03	0.97
100	0.01	1.32	0.02	1.36	0.02	0.99
110	0.01	1.33	-	-	0.02	1.01
120	0.01	1.33	-	-	0.02	1.02
130	-	-	-	-	0.02	1.04
140	-	-	-	-	0.02	1.06
150	-	-	-	-	0.01	1.07

Table 5. A comparison of the total yield obtained at various values of the microwave power used.

Distiller volume	700W	560W	420W
500mL	0.99	1.09	0.86
1000mL	1.12	1.14	1.15
2000mL	1.33	1.36	1.08

yield amounts to 1.36 % (Table 5). The extraction times at all power values studied show significant differences, namely 120 min, 100 min, and 150 min at 700 W, 560 W, and 420 W respectively (Table 4).

The study shows that the increase of the ratio of the distiller volume and that of the microwave cavity clearly increases the yield of extraction. The feed-to-solvent ratio of 0.02, 0.04, and 0.08 results in yield of 34 %, 25 %, and 34 %, respectively. This is caused by the rapid heating of the immersed material near the expanded wall and the higher pressure inside the plant material. The increasing microwave power ratio leads to expeditious releasing of the essential oil [14, 27, 31 - 33].

Chemical analysis by GC-MS

The identification of the chemical constituents is carried out by calculating the retention indices and their comparing with the peaks of the Wiley and Adams library search data. Citronella is the predominant component of the essential oil of *Cymbopogon nardus* obtained by microwave hydrodistillation. The extracted oil contains *Citronella*, *Geraniol*, and *Citronellol* with comprised percent areas of 40.54 %, 17.39 %, and 15.88 %, respectively. This result is similar to those of other researchers studying the main components of *Cymbopogon nardus* [1, 34 - 36]. The percentages of Citronella are higher than those in case of Cymbopogon nardus from Malaysia, northeast Thailand, Egypt, Malaka [35 - 38]. The differences of type of the components reported by the other researchers are probably due to the different climatic conditions of the plant growth region.

Table 6. Chemical compounds contained in the essential oil of dried leaves of *Cymbopogon nardus* obtained by microwave hydrodistillation.

Chemical	% of total	Quality
Citronella	40.54	98
Trans-Geraniol	17.39	86
β-citronellol	15.88	98
d-limonene	1.60	99
Isopulegol	0.35	98
cis-2,6-Dimethyl-2,6-octadiene	0.98	97
Eugenol	0.45	98
Delta-3-Carene	0.46	96
α.-cubebene	1.84	95
Delta.-cardinene	0.80	98
Torreyol	0.85	97
Naphthalene	3.95	96

CONCLUSIONS

Understanding the microwaves effect on the extraction of aromatic plants is important for the development of a large-scale extraction equipment. The study of the extraction of Cymbopogon nardus using microwave hydrodistillation shows that the increase of D/C ratio causes expansion of the distiller surface and provides even heating on the surface of the volume. Subsequently, the better distribution of the heat throughout the matrix increases the extraction yield. It might alternatively resolve the problem of the limited microwave penetration depth.

The highest amount of oil refers to the total amount of 1.42 % for 120 min extraction using 2000 mL distiller and 80 % of the microwave-power output ratio. The essential oils increase by 56 % from the lowest value of 0.91 % to that of 1.42 %. The chemical composition of the oil produced from *Cymbopogon nardus* plant from Pacet, Mojokerto, East Java contains mainly citronella, geraniol, and citronellol of comprised percent areas of 40.54 %, 17.39 %, and 15.88 %, respectively. In conclusion, an oil yield increase by a step up in the capacity of extraction of dried leaves of *Cymbopogon nardus* using a microwave equipment can be achieved. Future development of new geometries that match the microwave heating character will be required to increase the capacity of the extracted material.

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