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PARAMETER FOR SCALE-UP OF EXTRACTION CYMBOPOGON NARDUS DRY LEAF USING MICROWAVE ASSISTED HYDRO-DISTILLATION

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Large capacity in extraction using the microwave hydrodistillation method is always a challenge to do. The biggest obstacle in this method is the limitations of microwaves penetrating the material. Besides that, the high heat produced due to molecular heating simultaneously causes some components to degrade and reduce the quality of essential oils. The purpose of this research is to study the effect of the ratio of the sample plant (Cymbopogon nardus) to the solvent (F/S = 0.08, 0.16, 0.24 g/mL), as well as the ratio of volume distiller to volume microwave cavity (D/M = 0,04, 0,08 v/v) to the yield and quality of Citronella Oil produced. The experimental lead to the conclusion that the ratio of the sample plant to the solvent (F/S) of 0.24 g/ mL is the maximum for extraction of Citronella Oil. The ratio of volume distiller to volume microwave cavity (D/M) 0,08 v/v provides a higher yield compared to (D/M) 0,04 v/v with a longer extraction time. The qualitative analysis by GC-MS shows that the Citronella is the main component of Citronella Oil. The composition showed significant differences in the essential oil content of different ratio of volume distiller to volume microwave cavity (D/M). The ratio volume distiller to volume microwave cavity (D/M) 0,04 v/v gave 21 component with the highest Citronella percentage (40.54%). Compared to (D/M) 0,08 v/v gave 27 component with the Citronella percentage (18.64%). The results showed that different volume of distillers showed different yields and components.

Key words: Volume distiller, Microwave power, Feed to solvent (F/S), Cymbopogon nardus, GC MS

INTRODUCTION

For a century, aromatic oils have grown extensively and had a high economic value. The widespread use of essential oils in various products has caused an increase in the demand for essential oil. . Citronella Oil is one of the essential oils that have high commercial value. Citronella Oil traditionally used as an anti-insect and spraining. Its also used in products such as body care, the aroma of food, medicine, and sanitation. (De Toledo et al. 2016; Manvitha and Bidya 2014; Avoseh et al. 2015).

Citronella Oil extracted from one of the aromatic plants of the genus Cymbopogon nardus (L) Rendle of the Poaceae family. C.nardus (L) Rendle is a clumped plant, with leaves shaped like a downward curved ribbon with a width between 0.5 - 1.5 cm and a length of 50 to 100 cm. The leaf bones are parallel and in the middle with a non-woody and short ribbed stem. This plant is a perennial plant and widely grown in Asia and Southeast Asia.

One method used to extract this oil is hydrodistillation. This method is done by immersing all plant samples into water solvents, then heating. Heat flows by convection and increases the temperature of water solvents. Water solvents around the plant samples will damage the cell wall and oil bag in the plant matrix and then come out simultaneously. Evaporation will occur after the boiling point of the water and oil mixture is reached. Furthermore, steam is flowed in a cooling condenser to separate oil from water.

Microwave Hydro-distillation

In the last few years, microwaves used in the extraction of essential oil. The method is known as Microwave Hydro-distillation (MHD). Microwaves are electromagnetic waves which are at frequencies between 3-300GHz with wavelengths between 0.01 m - 1 m. (Pozar 2005). However, in the industrial world and the frequency of treatment that is permitted is in the range 0.915 - 2.45 GHz. (Chemat and Cravotto 2013a). MHD works by utilizing the response of polar (water) compound to microwave radiation at a frequency of 2.45 GHz. At this frequency, the microwave absorbed by the material and converted to heat. Microwaves are emitted by magnetrons and absorbed by water molecules (as solvents) and cause simultaneous molecular ion and ion conduction rotation (Thakker, Parikh, and Desai 2016). The ability of the material to absorb energy and turn it into heat is called loss tangent (tan δ) which is expressed by the tan equation $\tan \delta = \varepsilon''/\varepsilon'$ where ε'' is dielectric loss and ε' is the dielectric constant. Dielectric loss is the ratio value where electromagnetic radiation is converted into heat, while the dielectric constant is the ability of molecules to be polarized by electromagnetic fields. (Flórez, Conde, and Domínguez 2015). With two movements quickly and almost simultaneously this increase in water temperature will take place in a short time (volumetric heating). (Petigny et al. 2014; Nitthiyah et al. 2017; Stefanidis et al. 2014).

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Water which is a natural content found in plants reacts to microwave radiation. The fundamental difference between heating water in plants versus solvent water lies in the effects of microwave radiation. The movement of water molecules in plants works to damage cell walls and oil bags in the plant matrix. Meanwhile, the movement of water molecules in solvents accelerates heating and evaporation. The two processes that take place simultaneously will increase the speed of extraction. (Chemat and Cravotto 2013b; Petigny et al. 2014).

However, the biggest obstacle in this method is the limitations of microwaves penetrating the material. The depth of penetration is defined as the depth into the plant matrix at which the power density has decrease to about 37 % of its surface value.(Nitthiyah et al. 2017). Thus, after passing the depth of penetration, the microwave loses its heating effectiveness. The limitations of microwave penetration distances cause significant differences in the heating distribution of objects of different diameters. (Z. Zhang, Su, and Zhang 2018; H. Zhang and Electric 2017; Brodie 2008). Experimental results using spherical objects indicate that the highest temperature on an object with a diameter <10cm was at the center of the ball. Whereas, on the ball object with a diameter> 10cm the highest temperature was between the center of the ball and the outer wall of the ball. The limitations of microwave penetration depth can be a cause of a decrease in the effectiveness of heating.

The difference in heat distribution due to changes in object diameter has been known by many researchers before. But as far as the researchers know, the effect of changing the diameter of the object of heating in the extraction of plants has never been observed. This research method was carried out experimentally by comparing the effect of microwave extraction parameters on the distiller diameter which was different from measuring extracted oil (yield) and extraction time. In the future, the results of this study can facilitate the scale-up of the extraction of microwave-based aromatic plants.

MATERIAL AND SAMPEL PREPARATIONS

The Cymbopogon nardus (L) Rendle, as a sample, has been investigated for its pharmacological Potential (De Toledo et al. 2016). Sample of C.nardus was collected from Pacet, Mojokerto East Java at an altitute of about 400-600 above sea level. The plants material was grown naturally and harvested after eight months. Harvesting was done by cutting the leaf base and leaving stump remans at the base. To minimize drying during the delivery and storage period effects to the plants were minimized by splashing water. Leaves ware separated from plants and drying in an open space avoiding a direct sun for a five day. The dried leaves were cut into 5 mm lengths. Moisture content of the sample is measured using a moisture meter probe a moisture meter probe by range 2% - 60%, with accuracy 1%.

Water solvent was used since water molecules have a high dipole moment, and strongly absorb microwave (Eskilsson and Björklund 2000). Furthermore, the polarity of water decreases due to increasing temperature. At the range of 100oC and the critical temperature, the increased thermal agitation will reduce the strength of hydrogen bonds. These are leading to dielectric constant value similar to those of organic solvents (Flórez, Conde, and Domínguez 2015).

METHODS

Ratio feed to solven (F/S)

The ratio of feed to the solvent (F/S) is the ratio where the number of samples compared to the solvent. F/S is expressed in percent by.

$$F/_S = \frac{sample}{solvent} x100$$

Distiller to the volume of the microwave cavity (D/M)

A volume distiller comparison with microwave cavity (D/M) is the ratio of distiller compared to the microwave cavity. D/M is expressed in percent by

$$D/_M = \frac{Volume\ distiller}{Volume\ microwave\ cavity} x100$$

Microwave Power

Microwave power represent electrical energy that is converted into electromagnetic waves by magnetrons and stated with percent microwave power. Microwave power 100% refers to 60 seconds of radiation emitted, while 80% and 60% were 48 and 36 seconds respectively. The power used was 700W (100%), 560W (80%) and 420W (60%).

Microwave Assisted Hydro-distillation

Samples soaked into the water for 40 minute. Distiller that was placed inside domestic microwave oven (25 L capacity). The number of samples compared to solvents (F/S) was 8%, 16%, and 24% respectively. The percent of samples compared to the volume of distiller to volume microwave cavity (D/M) was 4%, and 8% respectively. The samples were extracted by hydrodistillation and Clevenger apparatus (fig 1) . The maximum temperature in the distillery was the temperature of boiling water and at atmospheric pressure. The steam which came out from the extraction chamber was condensed using Clevenger-type apparatus and collected a vial bottle. The water in the extraction yield was removed using anhydrous sodium sulfate and cooled to 4°C. The extraction was repeated three times for each variabel. The extraction yield of Citronella Oil was calculated according to the equation given :

$$\text{yield of citronella Oil } (\%, w/w) = \frac{mass\ of\ extracted\ citronella\ Oil}{mass\ of\ extracted\ cymbopogon\ nardus} x\ 100$$

The microwave oven power levels was 1000 W, 250 v-50 Hz and bursting microwave radiation at a frequency 2.45 GHz. Microwave radiation was used at microwave power 100%, 80% and 60%.

GC–Mass Spectrometry Identification

Citronella oils composition was determined by gas chromatography coupled to mass spectrometry (GC–MS) analysis Instrument Agilent 6890 N Network GC System, Agilent J & W Capillary GC Column (30 m x 0.320 mm x 0.25 μm). The sample was shaken and taken 0.11 μL and injected into the instrument.. The conditions maintained were : carrier gas Alpha Gaz Helium Ultra pure; split 1:100; Injection temperature 280˚C; oven temperature programmed from 50 to 230˚C at 5˚C min^{-1} and from 230 to 280˚C at 10˚C min^{-1}. Identification of Citronella Oil components is displayed by retention time, qual, percent area and compared with Wiley version 7.0 database.

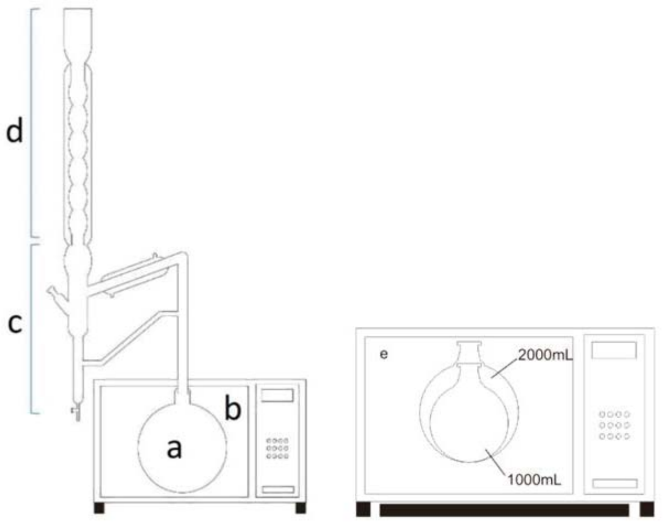

Figure 1: Microwave extraction equipment with extraction chamber (a); in microwave chamber (b); the steam discharged from the distiller was condensed in clevenger (c); with the cooling system (d); Diagram of the various volume distiller in the microwave chamber (e)

RESULT AND DISCUSSION

Effect of Microwave Power

Microwave heating temperature, strongly influenced by microwave power used in the system(Chemat et al. 2015; Desai and Parikh 2015; Ormeño, Goldstein, and Niinemets 2011). Three different microwave power inputs have used for the extraction process where the heating duration gives different total energy. The highest microwave power generates total energy 3.36 x 106 J. (700 J / sec x 60sec x 80min), while 80% and 60% produce 2.69 x 106 J and 2.02 x 106 J. In extracting aromatic plants, the use of high microwave power can increase extraction results (Chemat and Cravotto 2013a). High microwave power increases the temperature of the solvent and decreases the viscosity and surface tension of the solvent thereby facilitating solubility of the solute. Besides, the microwave ability to penetrate the plant matrix also increases. In this study, increased yield D/M 4% has compared to 8% and subjected to three different microwave power inputs was used for the extraction process (Fig. 2). The yield with different microwave power usage is shown in Fig. 3. Citronella Oil yield was measured based on oil extracted at the lowest microwave power of 60% (420W). The results show 80% of microwave power (560W) gives a significant yield increase of up to 15% on D/M 4%. While D/M 8% yield increased up to 13% occurs at 100% microwave power (700W).

Effect of feed-to-solven ratio (F/S)

Effect of the weight of plant sample to solvent (F/S) ratio on the extraction of Citronella oil by microwave hydrodistillation at the different volume distiller has to be studied because it is related to load factor of the distiller to obtain maximum yield.

Comparison of the ratio feed-solvent (F/S) in fig.3 has shown 4%, 16%, and 24% for various microwave power. By increasing the ratio of feed to solvents 4% to 16%, the yield increases from 1.07% to 1.28% or increased by 20%. However, increasing the F/S ratio to 24% increases the yield by 16%. Comparison of yields at 8% D/M ratio increases yield below 20% because of non-uniform distribution and exposure to microwaves (Sparr Eskilsson and Björklund 2000). However, an increase in the D/M ratio from 4% to 8% quite positively increase an extraction yield.

Effect of Extraction Time

In microwave extraction the duration of heating extraction is a factor to be considered. Increasing the time of microwave radiation can increase extraction yield. However prolong time, gradually decrease extraction yield. Fig. 4 shows yield from Cymbopogon nardus at different time. The highest extraction yield was obtained in 30 minutes although the maximum extracted oil achieved at 80 minutes of extraction. More than 80% of the oil was extracted at the beginning while the remaining 20% was 50 minutes later. This experiment confirmed the Fick's second law of diffusion takes place. With the length of extraction time, the solute concentration of the plant matrix in the solvent will reach equilibrium. Indeed, extending the extraction time will not significantly improve the results. The yield ratio between D/M 4% and 8% show, that to get the same yield, longer extraction time is needed in Fig. 5.

Chemical Composition

The components of the Citronella Oil are identified by GC-MS. The extract obtained by hydrodistillation on the ratio volume distiller comparison with microwave cavity (D/M) 4% (v/v) contains 21 components, while that the ratio volume distiller comparison with microwave cavity

(D/M) 8% (v/v) has 27 components. The GC-MS analysis results are listed in Table 1. The quality of the Citronella Oil obtained is determined by the content of Citronella, Geraniol and Citronellol as the major compound of Citronella Oil (Avinash Singh and Kumar 2017; De Toledo et al. 2016; Chanthai et al. 2012; Hamzah et al. 2014).

The content of Citronella in the extract obtained by the ratio volume distiller comparison with microwave cavity (D/M) 4% (v/v) amounts to 40.54 %, Geraniol 17.39% and, Citronellol 15.88%. While that in the extracted obtained by the ratio volume distiller comparison with microwave cavity (D/M) 8% (v/v) Citronella 18.64%,Geraniol 33.65% and, Citronellol 0.86%.

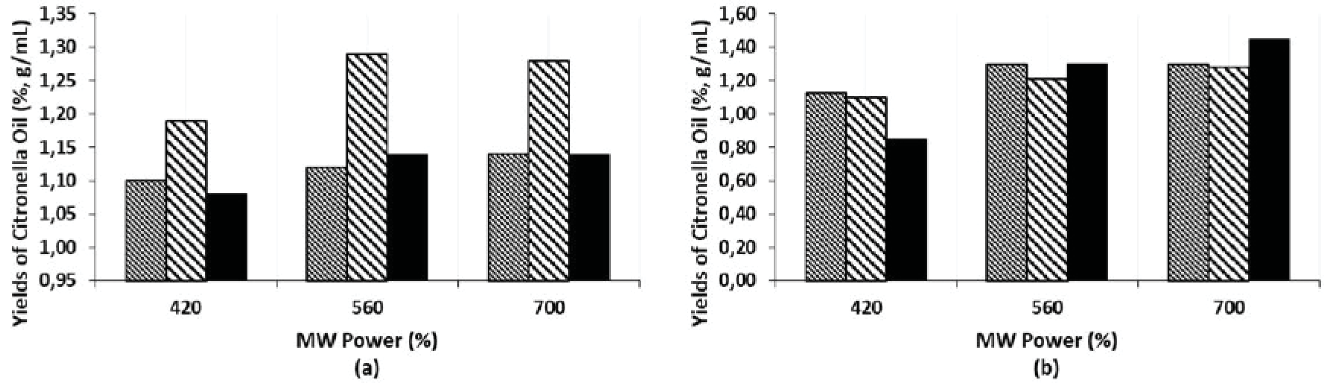

Figure 2: Efek of microwave power usage on the yield of Citronella Oil (mass of citronella Oil /mass of extracted Cymbopogon nardus) at 80 minute extraction time, with ratio volume distiller comparison with microwave cavity (D/M) (a) 4% (v/v) and, (b) 8% (v/v)

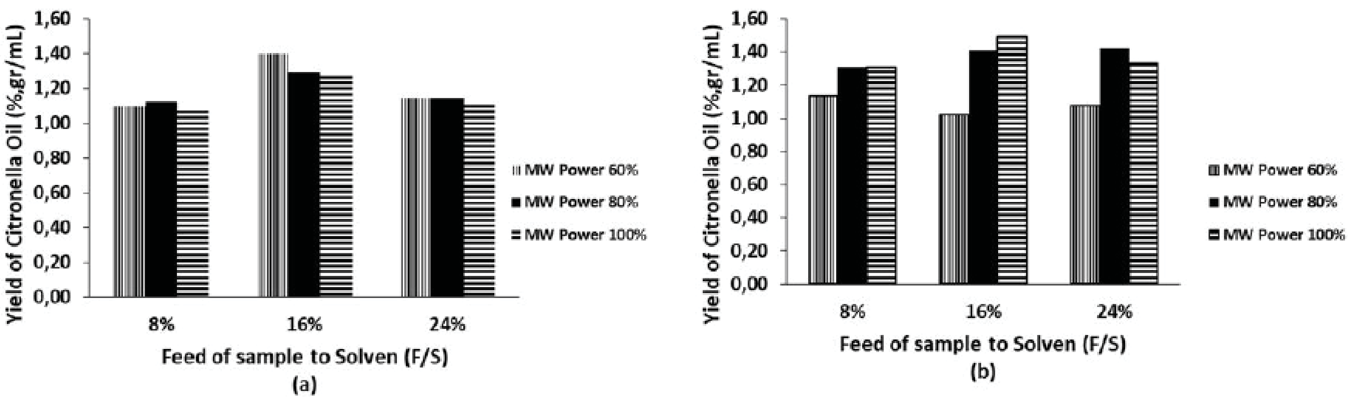

Figure 3: Efek of feed to solvent ratio (F/S) on the yield of Citronella Oil (mass of citronella Oil /mass of extracted Cymbopogon nardus) at 80 minute extraction time, with ratio volume distiller comparison with microwave cavity (D/M) (a) 4% (v/v) and, (b) 8% (v/v)

It is obvious that the main content obtained by the volume distiller comparison with microwave cavity (D/M) 4% (v/v) is higher when compared by the volume distiller comparison with microwave cavity (D/M) 8% (v/v). The differences strongly influenced by the heating time. Some components may be very easily broken down due to high heating. (Sadgrove and Jones 2015; Ashutosh Singh et al. 2014).

CONCLUSION

From these preliminary results, microwave energy used was found to be predictive of increasing yields extraction Cymbopogon nardus samples exposed to 2.45-GHz microwave when ratio F/S and time extractions were held constant. Microwave power also applied to the different ratio D/M. The effective volume-to-microwave-chamber (D/M) ratio increases yield at high microwave power for these microwave systems and distiller dimensions. These laboratory experiments provide sufficient justification to begin industrial scale-up of microwave energy as a tool to large capacity extraction. We propose that the microwave power ratio for extraction between 80 and 100 percent at 1000W electric power. Extraction times can be carried out in 30 to 50 minutes with an 8% D/M ratio and 24% F/S ratio.

In conclusion, increasing oil yield by a step up in the capacity of extraction of dried leaves Cymbopogon nardus using microwave equipment could be done. Additional experiments in microwave ovens with larger-size distiller are planned to verify the efficacy of these energy-to-volume distiller ratios.

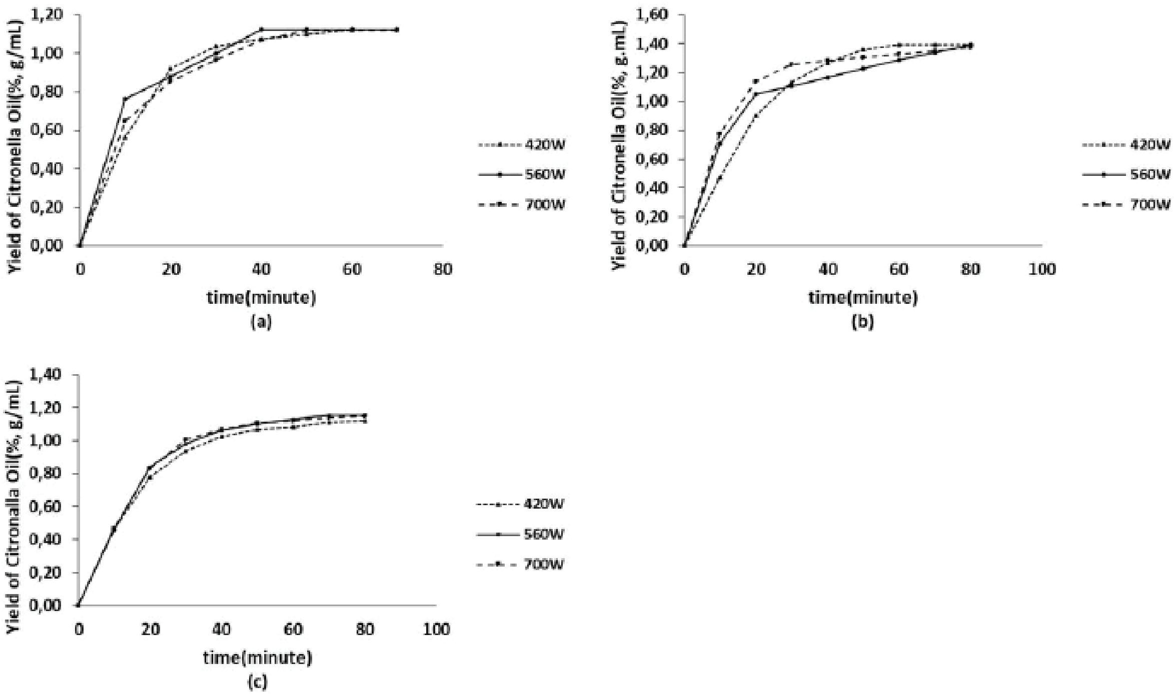

Figure 4: Accumulated on the yield of Citronella Oil (mass of citronella Oil /mass of extracted Cymbopogon nardus) at 80 minute extraction time, with ratio of volume distiller with microwave cavity (D/M) 4% (v/v). The microwave power was 420W, 560 W and, 700W. The ratio of Cymbopogon nardus leaves to water(F/S) was (a) 0.08 g mL (8%), (b) 0.16 g mL (16%) and, (a) 0.24 g mL (24%)

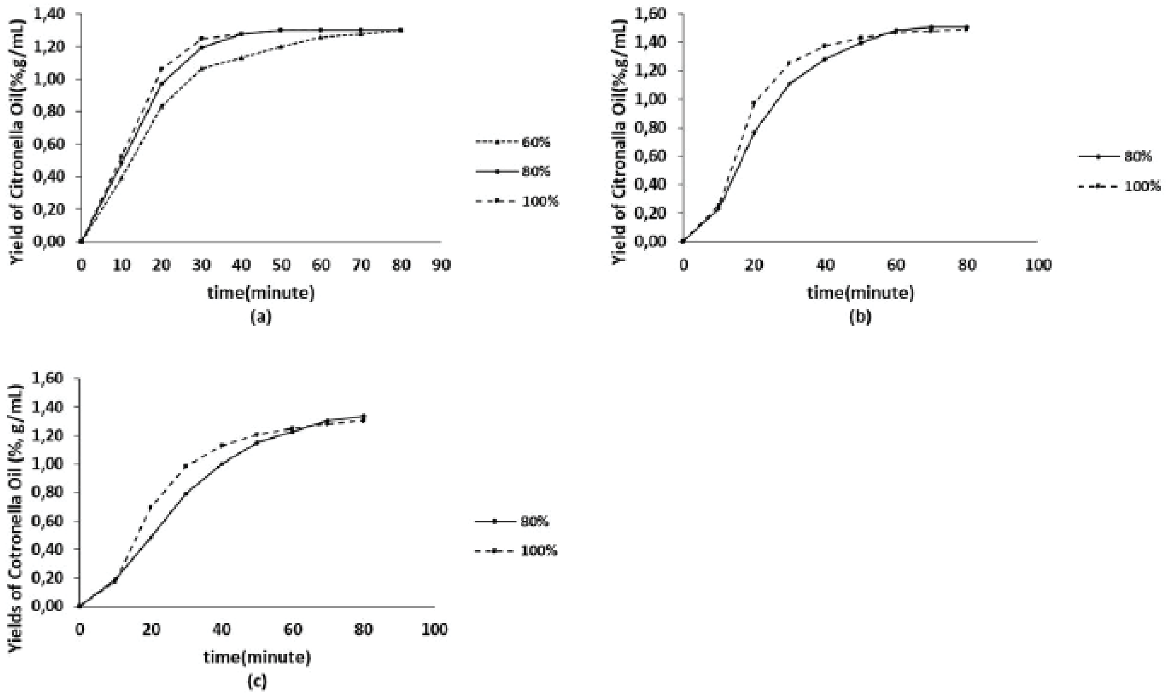

Figure 5: Accumulated on the yield of Citronella Oil (mass of citronella Oil /mass of extracted Cymbopogon nardus) at 80 minute extraction time, with ratio of volume distiller with microwave cavity (D/M) 8% (v/v). The microwave power was 420W, 560 W and, 700W. The ratio of Cymbopogon nardus leaves to water(F/S) was (a) 0.08 g mL (8%), (b) 0.16 g mL (16%) and, (a) 0.24 g mL (24%)

Table 1: Composition of Citronella Oil extracted by hydrodistillation the ratio volume distiller comparison with microwave cavity (D/M) 4% (v/v) and 8% (v/v)

Component	Retention time (min)	Volume distiller to microwave cavity (D/M) 4% (% of Total)	Qual (%)	Retention time (min)	Volume distiller to microwave cavity (D/M) 8% (% of Total)	Qual (%)
Butanal	1,366	9,19	91	-	-	
Cyclopentane	1,47	0,38	91	-	-	
dl-Limonene	8,58	1,62	99	5,50	0.98	98
Cyclofenchene	10.06	0,59	93	-	-	
Isopulegol	10,85	0,35	98	-	-	
Citronella	11,08	40,54	98	8.63	18.64	96
β-Citronellol	12,28	15,88	98	-	-	
Trans-Geraniol	12,69	17,39	86	11.38	1.65	87
Cis-2,6-Dime-tyl-2,6-octadiene	14,04	0,94	97	-	-	94
Phenol	14,12	0,45	98	-	-	
Δ 3-Carene	14.46	0,46	96	-	-	
Not identified	15.00	0,82	92	12.74	1.55	93
Not identified	15.80	0,59	93	13.40	0.85	94
Not identified	-	-	-	14.49	1.55	90
Not identified	-	-	-	14.99	1.73	64
Copaene	15,96	0.40	93	-	-	
α-Cubebene	16.21	1.84	95	-	-	
Δ Cadinene	16.30	0.80	98	13.19	0.39	93
Cyclohexane-methanol	16.61	1.30	90	18.96	0.92	47
Naphthalene	16,95	2,47	96	13.71	0.95	98
Torreyol	17.70	0,85	97	15.26	2.27	91
Naphthalene	17.85	1.48	91	13.86	6.02	97
Bis-phthalate	26.04	1.66	90	23.47	4.89	83
Z-Citral	-	-	-	10.01	0.86	95
1,6-octadien-3-ol, 3,7-dimethyl-(+/-) Geraniol	-	-	-	10.78	30.55	70
Geraniol	-	-	-	11.55	1.44	60
Eremophilene	-	-	-	11.75	2.42	64
2,6-octadien-1-ol, 3,7-dimethyl	-	-	-	11.90	0.74	90
β-elemene	-	-	-	12.17	4.87	99
Trans-Caryophyllene	-	-	-	12.51	5.78	99
β-Cubebene	-	-	-	12.61	0.34	96
α-Humulene	-	-	-	12.92	1.59	97
Trans-β-Farnesene	-	-	-	13.07	0.53	96
Germacrene	-	-	-	13.29	2.49	97
1H-Cyclo-propa[a] naphthalene	-	-	-	13.57	1.18	96
Guaiol	-	-	-	14.82	1.53	93
Neophytadiene	-	-	-	17.34	0.24	99

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