



Essential Oil Extraction from Citronella (*Cymbopogon nardus* (L.)) Using *Solvent Free Microwave Extraction* Method (SFME)

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Abstract. Indonesia is one of the producing countries essential oils, essential oils are also a commodity that can generate foreign exchange for the country. Therefore, essential oils receive special attention from the Indonesian government. Indonesia generate 40-50 types of plants that produce essential oils and are traded in the world. Extraction using a microwave with the basic mechanism of microwave heating involves stirring polar molecules or ions that oscillate due to the influence of electric and magnetic fields called dipolar polarization. From the results of physical analysis, it can be seen that citronella essential oil obtained using the Solvent Free Microwave Extraction (SFME) method has met the standards and quality of citronella oil based on SNI 06-3953-1995, according to the parameters of color, specific gravity, and solubility in water. 80% ethanol shows citronella oil with good quality. The results of GC-MS (Gas Chromatography-Mass Spectrometry) analysis on the extraction of citronella essential oil using the Solvent Free Microwave Extraction (SFME) method obtained 2 components, namely citronella and geraniol, with citronella percentages of 8.64% and 7.53%. Optimal operating conditions for the extraction of essential oils from citronella raw materials using the Solvent Free Microwave Extraction (SFME) method.

Keywords: *Extraction, Lemongrass, Microwave, GC-MS (Gas Chromatography-Mass Spectrometry)*

1. Introduction

Indonesia is a country that produces essential oils, essential oils are also a commodity that can generate foreign exchange for the country. Thus, essential oils receive special attention from the Indonesian government. Indonesia produces 40-50 types of plants that produce essential oils and are traded in the world. Until now, Indonesia has only produced several essential oils such as: clove essential oil, ylang leaf essential oil, patchouli leaf essential oil, vetiver essential oil, nutmeg essential oil, eucalyptus essential oil, citronella essential oil and

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sandalwood essential oil. In Indonesia, there are 6 types of essential oils, the most prominent of which are: nutmeg oil, vetiver oil, patchouli oil, eucalyptus oil, clove oil and citronella oil.

Lemongrass (*Cymbopogon nardus* (L.)) is one of the essential oil-producing plants from the Poaceae family and was known in Indonesia before World War II. Citronella contains about 1-2% essential oil on a dry basis and the composition of the essential oil depends on the diversity of treatments, habitats, and plant genetics [1]. According to Citronella oil is the name of citronella essential oil in the trading world. Citronella essential oil is included in one of the essential oil commodities that have considerable prospects among other essential oils [2].

The citronella plant has a characteristic lemon scent due to the main content of citronella, which is citral. Citral is a combination of netral and geranial isomers, usually used as raw material for ionine, beta carotene, and vitamin A products. Lemongrass essential oil has also been shown to have high anti-oxidant, anti-bacterial and anti-fungal properties [3].

The process of extracting essential oils usually uses the conventional method, namely the hydrodistillation method, previous studies have shown that this distillation method takes a long time and also requires a lot of solvents to get yield results, this is less efficient in terms of time and energy and less friendly to the environment [4]. Therefore, a method was developed in the process of extracting essential oils, namely by using the Microwave Assisted Extraction (MAE) method. The Microwave Assisted Extraction (MAE) method consists of Microwave Assisted Hydrodistillation (MAHD), Microwave Steam Distillation (MSD), Microwave Steam Diffusion (MSDf), and others.

Based on the above method, further development of the next Microwave Hydrodistillation (MHD) method was carried out, namely the Solvent-Free Microwave Extraction (SFME) method. In this method, the extraction process is carried out without using any solvent and also utilizing heat from the microwave [5]. This method also combines microwave heating and distillation with atmospheric pressure. The principle of this method is that it does not use water or organic solvents, so that the extraction process uses the water content contained in the plant, so during the extraction process the raw materials to be extracted will not come into contact with chemicals [4].

Based on the description above, this research uses citronella to extract essential oils using the extraction method, namely Solvent-Free Microwave Extraction (SFME), which has never been done before, so this research was carried out using the Solvent-Free Microwave Extraction (SFME) and analyzed the quality of citronella essential oil produced based on the

standard of SNI 06-2386-2006.

2. Materials and Methods

2.1. Materials

The materials used in this study were citronella leaves with a water content of 48-52% and 80% ethanol. For the schematic of the tool in the Solvent Free Microwave Extraction method, the main tool consists of a microwave. The specifications of this research equipment are Microwave, 100 ml beaker glass, 250 ml beaker glass, Vial bottle, Spatula, Analytical balance, 1000 ml round bottom flask, 1000 ml wide 2nd round flask, Oven, Clamps, Stative, Gass Chromatography-Mass Spectrometry (GC-MS) and Scanning Electron Microscope (SEM).

The research was carried out at the Basic Chemistry Laboratory and the Bioprocess Laboratory of the Chemical Engineering/Engineering Study Program, Department of Mechanical Engineering, Faculty of Engineering, University of Jember. Research activities are carried out approximately from November 2020 to January 2021.

2.2. Material preparation

The first step the raw material in the form of citronella leaves is taken fresh from the field, then the citronella leaves are cleaned of dirt that sticks so as not to interfere with the extraction process, then the citronella leaves are cut into sizes of 2-3 cm.

2.3. Extraction using the Solvent Free Microwave Extraction (SFME) method

The extraction process begins by weighing the raw materials according to the variables then installing the extraction tool then inserting the raw material for citronella into a distillation flask, for the next step to drain the water in the cooling water system then insert the flask into the microwave, after the flask is inserted the next step is to turn on the microwave and adjust the microwave power according to the variable, then record the distillation time starting from the first drop of distillate out, then stop the extraction process after the specified time according to the variable and for the last step to store the essential oil in a vial.

3. Result and Discussion

This study used citronella raw material obtained from Kemuning Lor Village, Arjasa District, Jember Regency and has been determined according to the variable, namely in fresh condition. Here as seen in the Figure below:



Figure 1. Fresh scented lemongrass plant

The variables used in this study are the ratio of the mass of raw materials to the volume of the distiller, microwave power and extraction time. The ratio of the mass of raw materials in this study is 0.05; 0.10 and 0.15 g/ml in fresh condition and put in a distiller flask with a volume of 1000 ml, the size of cutting the material is 2-3 cm long because the citronella leaves are surrounded by oil glands, oil pockets and vessels so that if not cut it will cause oil is not extracted optimally. The selection of this material mass ratio variable aims to predict yield results. For microwave power in this study using power 300 W, 450 W, and 600 W. The choice of microwave power in this extraction is due to the influence on the amount of heat energy that will be received by the raw materials [7].

Extraction of citronella essential oil in this study used the Solvent-Free Microwave Extraction (SFME) method from fresh raw materials. The yield produced using this method is large due to the influence of the water content in the fresh ingredients. This is because the extraction with fresh raw materials has a much smaller amount of water in the distiller. a small amount of water will accelerate the increase in temperature and with this rapid increase in temperature, it will accelerate the opening of the oil glands and cause a faster rate of increase in yield.

Solvent-Free Microwave Extraction method is an extraction method without the use of solvents by utilizing microwaves as a heater. the extraction process of the Solvent Free Microwave Extraction method occurs synergy between mass transfer and heat transfer from inside and outside due to internal overheating so that the extraction process is faster. In this method, the water contained in the material is refluxed into a distiller flask using a clevenger.

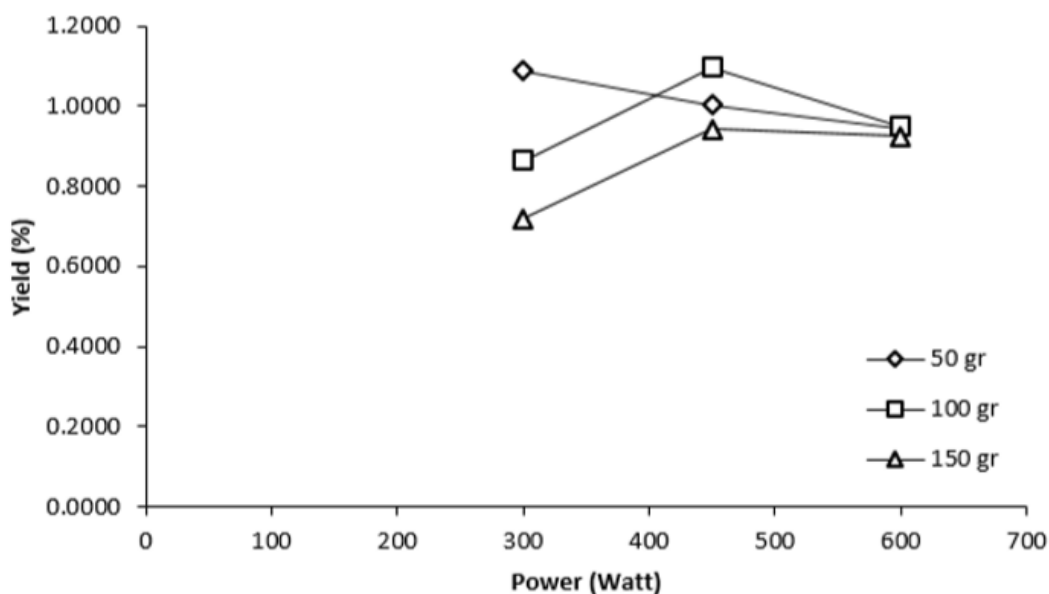


Figure 2. Graph of the effect of microwave power on oil yield fragrant lemongrass leaves

Power is the amount of energy delivered per unit time. In the extraction process, power has a major influence on the yield of citronella oil that will be produced. Microwave power is closely related to process temperature, where the greater the power to be used, the system temperature in the extraction process will increase faster [7]. The effect of power on temperature lies in the increase in temperature. The greater the power, the faster the temperature rise that occurs in the extraction process.

Based on Figure 2, it can be seen that the highest yield is at 450 watts at a mass ratio of 0.10 g/ml. Similar to research [9] on vetiver extraction, the highest yield value was obtained when the power was 450 watts in fresh raw material conditions. This is because the power is stable (power is not too low and high, so if low power causes heat transfer to be hampered and difficult to enter into the material, while for high power it can cause the material to burn quickly due to high heat transfer heat and damage the glands oil), then with a stable power can result in the material can be extracted properly. In this parameter, it can be seen that if the greater the microwave power used, the polar molecules in the material when exposed to microwave radiation will experience a faster rotation (oscillatory motion and collide with each other) and produce heat energy (*heat*) so that the target molecule can be extracted. out of the material. Yield reduction occurred at 300, 450 and 600 watts at a mass ratio of 0.05 g/mL. The cause of the decrease in yield is due to the small mass of raw materials used, while the small power also affects the small energy transfer in the sample and causes the yield to be not optimum due to material degradation. Supported by research, namely the extraction of pomelo peels using the

SFME method at a power of 300 watts and 450 watts the yield decreased by 5.3% due to damage to the oil glands at higher microwave power. Power that is too high can also cause raw materials to dry faster. According to [7] the water content in the plant also has an effect, so the higher the power used, the faster the boiling point. In this study, the optimum yield was obtained at 300 watts of power and 450 watts of power with a mass ratio of 0.10 g/ml. The water content in the plant also has an effect, so the higher the power used, the faster the boiling point. In this study, the optimum yield was obtained at 300 watts of power and 450 watts of power with a mass ratio of 0.10 g/ml. 2008) the water content in the plant also has an effect, so the higher the power used, the faster the boiling point. In this study, the optimum yield was obtained at 300 watts of power and 450 watts of power with a mass ratio of 0.10 g/ml.

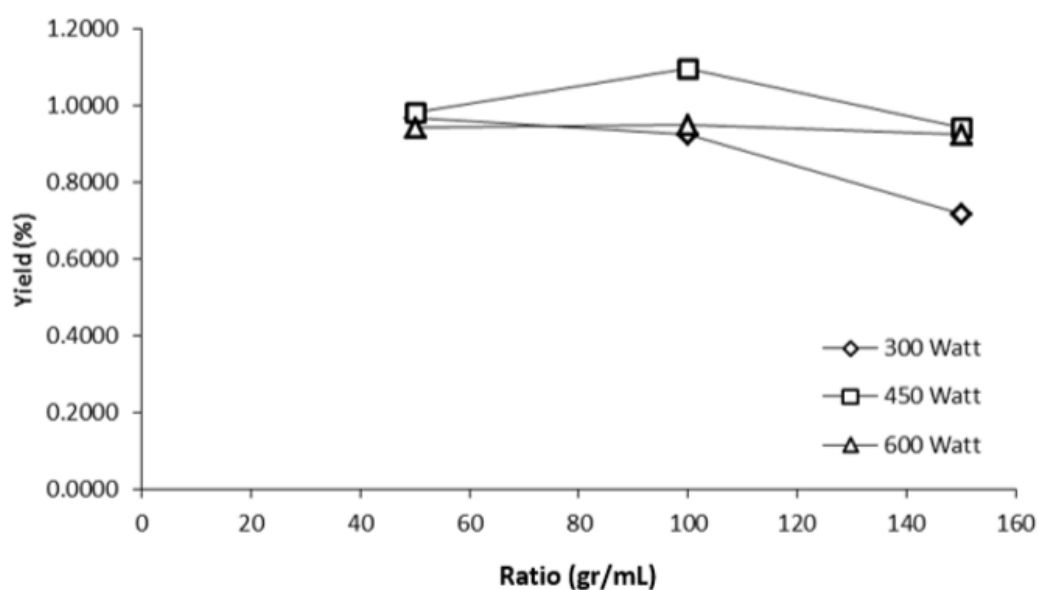


Figure 3. Graph of effect of ratio between material mass standard with distiller volume (f/d) against yield citronella oil

Extraction of citronella oil by solvent- free microwave extraction method using a mass ratio of 0.05; 0.10; 0.15 g/mL and for each variable will be placed in a distiller flask with a volume of 1000 mL. The effect of material mass per distiller volume (F/D) on yield can be seen in Figure 3.

Based on Figure 3, the optimum ratio is found at 0.10 g/mL. This is supported by research on the extraction of essential oil of eucalyptus leaves using the Solvent Free Microwave Extraction (SFME) method in fresh conditions, the optimum yield was obtained at a mass ratio of 0.10 g/ml. Furthermore, in the extraction of citronella essential oil, there was an increase in yield from a ratio of 0.05 gr/ml to 0.10 gr/ml at 450 watts of power. However, there

was a decrease in yield at a ratio of 0.15gr/ml. This is supported by research on the extraction of essential oils from stems, leaves, and peels of limes with the Solvent Free Microwave Extraction method on the effect of the ratio between the mass of raw materials and the volume of the distiller on the yield of fresh kaffir lime leaf oil. optimal at 0.25 g/ml, Broadly speaking, the yield increased from a ratio of 0.05 gr/ml to 0.25 gr/ml, but at a ratio of 0.3 gr/ml it decreased and was caused by several factors such as the amount of material in the distiller flask, the density of the material can cause the formation of pathways. “rat holes” steam which will affect the yield of essential oils, either increasing the yield or reducing the yield of essential oils. The material density factor is the ratio between the mass of the material and the volume capacity of the distiller flask. This can cause the steam generated by microwave heat to be difficult to penetrate in the material to carry the diffused oil molecules out of the material [6]. In addition, the ratio used relates to how dense (amount) of raw materials are included in the distiller flask, this causes the yield of citronella oil to decrease. For the lowest yield, based on Figure 2, which is 0.7181 at 300 watts of power, 150 grams of material and 60 minutes of time.

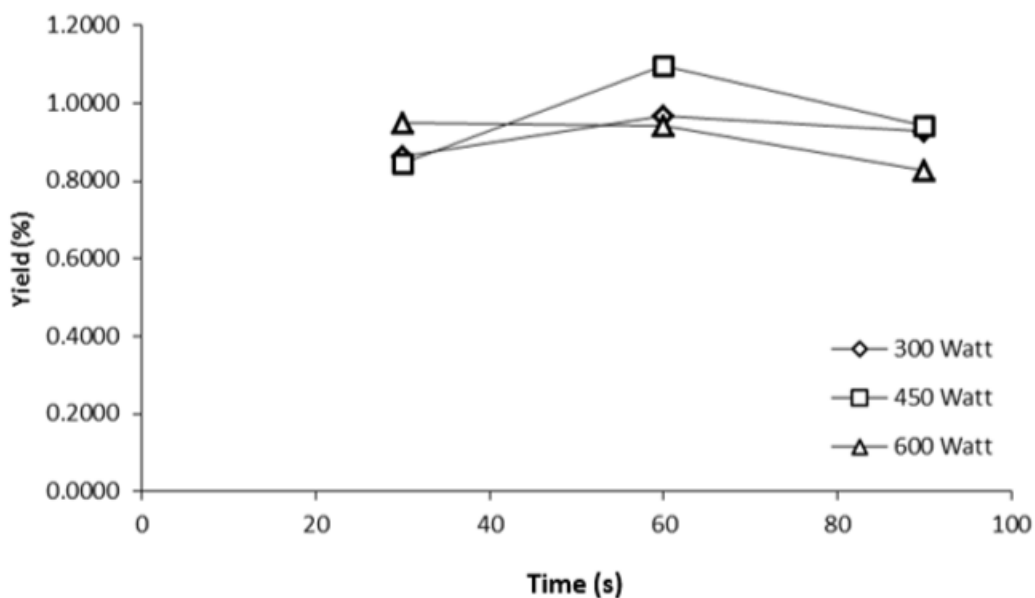


Figure 4. Graph of the effect of time on yield lemongrass oil

Figure 4. shows the relationship between extraction time and yield of citronella oil. The extraction of citronella oil using the Solvent Free Microwave Extraction method for 30 and 60 minutes, namely at 300 watts of 450 watts produces yields that tend to increase constantly with increasing extraction time. However, at 600 watts the yield decreases due to the high heat released causing the raw material to dry quickly. The lowest yield is at 90 minutes of 600 watts of mass 100 grams with a yield of 0.8259, this is because the high power and longtime cause

the raw materials to dry up to charred. According to with the longer extraction time, the increase in yield obtained becomes smaller or smaller.

The quality of essential oils is influenced by several factors such as raw materials, post-harvest handling, production and storage processes. Based on the results of this study, it is known that the quality of citronella essential oil is influenced by one factor, namely raw materials, the raw materials in this study are in different conditions, such as differences in material conditions from post-harvest to the extraction process, material conditions from fresh to slightly wilted. and this causes the content of the essential oil is different. From the results of the Gas Chromatography-Mass Spectroscopy (GC-MS) test, there are 84 components contained in the essential oil of citronella. There are two compounds with the highest % area, namely 6- octenal,3,7-dimethyl and geraniol, 6- octenal,3,7-dimethyl is another name for citronellal with a yield of 8.64% with a molecular weight of 154 and belongs to the class of oxidized monoterpenes. The second highest compound was geraniol with a yield of 7.53%, a molecular weight of 154 and was included in the oxidized monoterpene class.

In a previous study for the results of geraniol and sitonellal citronella essential oil using methanol as a solvent, namely 20.07% and 36.11%, respectively. These results are different due to several factors such as the method used and also the area of origin of the raw materials, such as weather factors and soil conditions during the citronella planting process. The GC-MS analysis test in this study used a random sample.

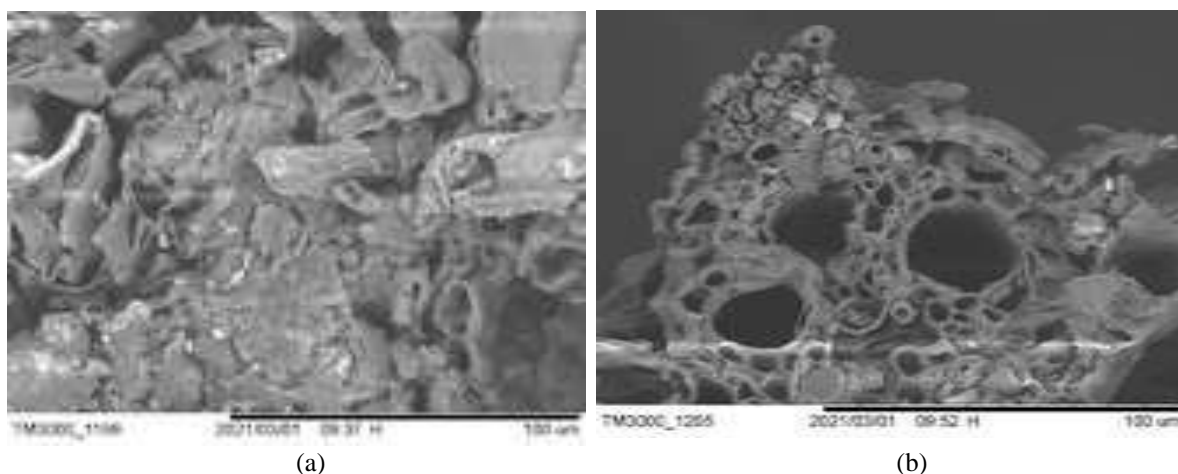


Figure 5. SEM results of citronella leaves with 1000 times magnification (a) before extraction (b) after extraction

4. Conclusion

The effect of microwave power, the ratio of the mass of raw materials to the volume of the F/D distiller and the resulting extraction time as well as the suitability of the extraction

results to SNI, namely the increase in yield occurs at a power of 300 - 450 watts, but decreases at 600 watts with a raw material ratio of 0.10 g /ml, the amount of raw materials contained in the distillation flask causes an increase in the amount of yield, namely in the ratio of 0.05 g/ml – 0.10 g/ml, but there is a decrease in the ratio of 0.15 g/ml due to the density of raw materials, time extraction tends to increase in the variable 30-60 minutes, but there is a decrease in the extraction time of 90 minutes, the results of the GC-MS analysis show that there are 2 main components of citronella essential oil, namely citronella and geraniol with an abundance percentage of 8.64% and 7.53%, the analysis of the physical properties of citronella essential oil has good quality because it meets the Indonesian National Standards, namely the parameters of color, specific gravity, and 80% alcohol solubility. The optimum conditions for extracting citronella essential oil using the Solvent Free Microwave Extraction (SFME) method are as follows, the optimal power obtained when using 450 watts of power with a yield of 1.096%, the ratio between citronella and distiller (F/D) obtained the optimum conditions at the mass ratio 0.10 gr/ml, with a yield of 1.0969%, the optimal time for the extraction process is 60 minutes, with a yield of 1.0873%.

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REFERENCES

- [1] Ranitha, M., AH Nour, ZA Sulaiman, AH Nour, and ST Raj. A comparative study of Lemongrass (*Cymbopogon citratus*) essential oil extracted by Microwave-Assisted Hydrodistillation (MAHD) and Conventional Hydrodistillation (HD) Method. *International journal of chemical engineering and applications* 5(2) (2014).
- [2] Sulaswatty, A., MS Rusli, H. Abhimanyu, and S. Tursiloadi. *Quo vadis citronella oil and its derivatives products*. Jakarta: LIPI Press 2019.
- [3] Ranade, SS and P. Thiagarajan. Lemon grass. *International journal of pharmaceutical sciences review and research* 35(2) (2015) 162-167.
- [4] Lucchesi, ME, Chemat, F. & Smadja, J. Solvent-free microwave extraction: an innovative tool for rapid extraction of essential oils from aromatic herbs and spices. *The Journal of microwave power and electromagnetic energy*, 39(3-4) (2004) 135-139.
- [5] Li, Y., Fabiano-Tixier, AS, Vian, MA & Chemat, F. Solvent-free microwave extraction of bioactive compounds provides a tool for green analytical chemistry. *TrAC Trends in analytical chemistry*, 47(6) (2013) 1-11.
- [6] Guenther, E, *The essential oil*, New: Van Nostrand Company Inc IV.1950.