Effect of Addition of NaCl Salt on Extraction of Essential Oil from Lemongrass Leaves by Microwave Hydro-Distillation Method

Fenni Suryanti, Lailatul Qadariyah, and Mahfud Mahfud*

Department of Chemical Engineering, Faculty of Industrial Technology and Systems Engineering, Institut Teknologi Sepuluh Nopember, Surabaya 60111, Indonesia

*** Corresponding author:**

email: mahfud@chem-eng.its.ac.id

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Abstract: *Essential oils are compounds extracted from plants and obtained by distillation. Indonesia has many kinds of plant commodities for essential oil production. Among the essential oils that have not been developed in Indonesia is citronella oil which can be extracted from the lemongrass plant this research, we develop Microwave Hydro-Distillation (MHD) method has several advantages over conventional distillation methods: shorter time, higher oil quality and yield. This research proposes to extract citronella oil from lemongrass leaves using the MHD method using aquadest and NaCl solution addition. Operating variables are extraction time (20–180 min), material size (0.5, 1.0, and 1.5 cm), feed-to-solvent ratio (0.1; 0.15; and 0.2 g/mL), and microwave power (300, 450, and 600 watts). The essential oil results are analyzed by GC-MS analysis, specific gravity, refractive index, and solubility. The results showed that it increased with extraction time followed by almost constant conditions, tended to decrease with increasing feed-to-solvent ratio, and increased yield with increasing microwave power. The results from the GC-MS analysis, the active substance content of geraniol was 46.61% and citronellal 5.62%. Additional salt in this method is a green and clean essential oil extraction.*

Keywords: *extraction; essential oil; lemongrass;* Cymbopogon nardus*; Microwave Hydro-Distillation*

INTRODUCTION

The variety of biological nature in Indonesia is famous and very abundant. There are essential oil plants that have not been able to be utilized as a whole. Indonesia has to be able to produce around 80 types of essential oil plants that can be traded in the world. Still, only a few kinds of essential oils can be produced by traditional refiners, including patchouli oil, citronella oil, clove oil, cananga oil, eucalyptus oil, sandalwood oil, fragrant root oil and citrus hystrix oil [1]. Essential oil is a high commodity requirement for perfume industries, cosmetics, pharmaceuticals, and food and beverage [2]. Producing primary and secondary products with price fluctuations resistance in the world trade area is highly valuable. However, essential oil production plants in Indonesia have not been adequately developed yet, so essential oil production cannot meet the quality and quantity requirements [3].

Lemongrass is a valuable plant. One of its usefulness is as an essential oil raw material. This plant is easy to cultivate and a significant opportunity for people who want to develop it [4]. Lemongrass plants have a significant potential to cultivate in Indonesia. Furthermore, Indonesia has this plant very abundant as a raw material. Many provinces in Indonesia produce essential oil, such as Nangroe Aceh Darussalam, Lampung, East Java, and West Java, with a total area of 3.492 hectares [5]. There are 2 kinds of variants of lemongrass leaves cultivated in Java, and one is *Cymbopogon winterianus* which is cultivated at West Java and one more is *Cymbopogon nardus* which is cultivated at East Java. They have different chemical compounds, respectively [6-7].

Essential oil extraction commonly uses a conventional method: water or steam distillation [8]. Essential oils are colorless to slightly yellowish and only

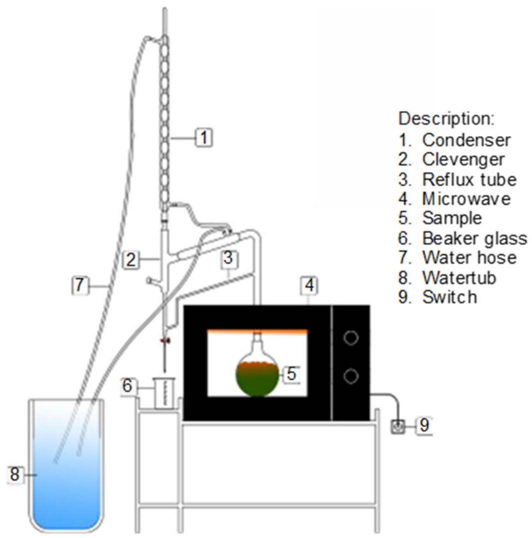

Fig 1. Schematic of MHD method

slightly soluble in water [9]. Extraction of lemongrass essential oil by hydro-distillation is known by people as 7–8 h extraction [10]. Essential oil product handling is also not optimal yet, causing less quality and low prices. The selection of a distillation system based on the leaves and stem of the plant as raw materials to produce higher yield, more efficiency, and avoid burnt leaves [11]. The lack of a system by traditional distillers (Low value of essential oil yield and quality) has to be overcome quickly by developing extraction technology by microwaves, one of which is by MHD method [12-13]. Microwaves are non-ionic electromagnetic waves below radio frequency and above infrared frequency waves, with a frequency distance between 300 MHz and 300 GHz. In the oven, microwaves work at a frequency of 2,450 MHz for general purposes and extraction applications for chemical analysis [14].

For the development of microwave technology, a medium is required to boost the solvent's dielectric coefficient so that it can be optimally absorbed and transformed into thermal energy to increase extracted essential oil and break down protected oil molecules [15-16]. Inorganic salts would cause more damage to the plant tissue's epidermal cells, making the essential oil easier for water vapor to extract and increasing the yield and separation effectiveness of the oil.

This research aims to study the effect of adding NaCl salt on the extraction of lemongrass leaves by the MHD method. This research will review the effect of microwave power, extraction time, the ratio between feed and solvent (F/S), and NaCl level toward extraction yield and oil quality to determine the best value conditions of lemongrass leaves extraction by microwave to produce citronella oil. NaCl salt is very effective in avoiding losses of the heat-sensitive components, especially the main components in essential oil, suit for a cleaner essential oil product to develop the modern essential oil industry [17].

■ EXPERIMENTAL SECTION

Materials

Lemongrass leaves are from *Cymbopogon nardus*, taken from Lumajang, East Java. The leaves have withered for 2×24 h to reduce moisture content and minced following the research variable. The research material is based on a dry basis [18]. We used the leaves part based on Feriyanto et al. [19], which yielded higher than stem part yields. To separate between essential oil and its solvent, used *n*-hexane ACS grade (95% pro analysis, CAS No. 110-54-3). NaCl solution made from NaCl 99%.

Instrumentation

The MHD instrumentation scheme was handled by a microwave oven (Electrolux EMM2308X) (Fig. 1), which has dimensions of 50 cm in length, 40 cm in width, and 40 cm in height. The power of the microwave ranges from 0 to 800 W. In this research, the power used was 150, 300, 450, and 600 W. Extraction flask volume 1 L as a glassware of material and solvent carried out the extraction process. Clevenger facilitates the extraction process so that while the process is in progress, solvents that have evaporated and condensed into the liquid phase can return to the extraction flask through the connection on the tool. In addition, the Clevenger is equipped with a Liebig condenser that helps the condensation process from vapors containing extracted oil. The instrumentation also has a Liebig condenser to maximize the cooling system.

Procedure

The MHD used a microwave oven. For the pre-treatment procedure, lemongrass leaves were withered for 2×24 h, minced by size according to the variables

(0.5, 1.0, and 1.5 cm), and stored at room temperature ± 30 °C. MHD research procedure is conducted by weighing lemongrass leaves according to the variables (30, 45, and 60 g). Put the weighed lemongrass leaves into the one-neck extraction flask, add 300 mL solvent according to the variables, and flow the water in the cooling system (condenser reflux and Liebig on the Clevenger). Turn on the microwave so that the extraction flask containing raw materials gets exposed to microwave radiation according to operating conditions and research variables. When the first drops come out from the Liebig condenser, it calculates the extraction time starting. Stop the extraction process according to predetermined time variables. The result is in the form of oil and solvent poured into the separator funnel and added *n*-hexane. The mixture between oil and *n*-hexane from the solvent was separated. Oil and n-hexane were separated. The essential oil was kept in a vial bottle at a temperature of 4 °C to prepare and analyze the essential oil obtained.

The experimental variables used in this study are as follows: Microwave power: 300, 450, and 600 W; material measure: 0.5 cm, 1 cm, and 1.5 cm; ratio F/S: 0.1, 0.15, and 0.2 g/mL; solvents: aquadest and NaCl 2% solution; extraction time: 180 min (sampling time every 20 min). Calculation of yield takes account of the moisture content of leaves according to the research by Chen et al. [20], and the yield of citronella oil can be calculated by Eq. (1):

$$\text{Yield}(\%) = \frac{\text{essential oil mass}}{\text{mass of leaves (dry basis)}} \times 100\% \quad (1)$$

Gas chromatography-mass spectrometry (GC-MS) analysis

GC-MS analysis results describe components contained and levels by peaks of components present in essential oil in detail [21]. The analysis was performed using GC-MS (AGILENT 6980N chromatography gas coupled with AGILENT 5973 inert mass spectrometry), carrier gas: Helium. The sample was injected at 250 °C inlet temperature with a split ratio of 1:50. The flow rate inside the GC column was held constant at 1 mL/min.

RESULTS AND DISCUSSION

Effect of Time on the Yield of Citronella Oil

The influence of time in the extraction of essentials is that the longer the extraction time, the more the amount of oil is obtained until a state of equilibrium when there is no increase in yield. It was called the optimal extraction time. In traditional distillations, the optimal extraction time 7 approximately h [22], while using a microwave only ranges from 2–3 h.

Effect of Power of the Yield of Citronella Oil

Power is the energy delivered per unit of time (J/s). In microwave extraction, power controls the amount of energy materials will receive to convert into thermal energy. This thermal energy conveys the movement of essential oil from a part of the plant to be extracted very well [23].

Microwave power must be carefully selected to minimize time consumption to obtain a temperature set without reaching excessive temperatures and overpressure. However, increased power with a longer microwave radiation time can lead to solvent loss due to evaporation. The variation of power used for the lemongrass leaves extraction process are 300, 450, and 600 W [24]. Fig. 2 shows the yield for citronella oil increases as long as power increases, at 300 W (1.394%) at power 450 W (2.110%), experiencing an increase in yields. However, at power 600 W (0.904%), the yield of essential oils decreased and tended to be lower than other variables.

Based on the graph, there is a tendency to increase the yield along with the increase in power. The more incredible energy received, the higher yield gets obtained because more energy is converted into the heat of

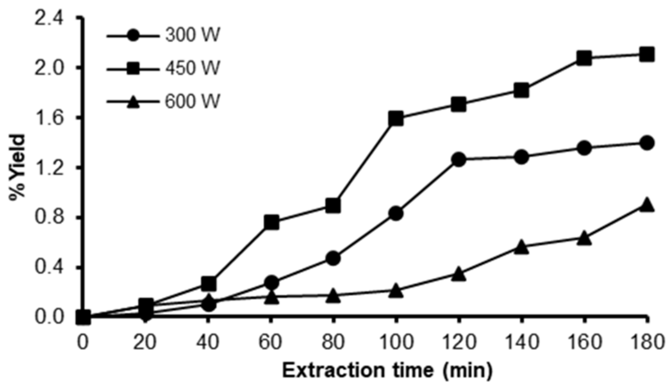

Fig 2. The oil yield function of time for different microwave power

extraction, increasing the extraction yield. The decreasing yield of 600 W caused by the power does not work at an optimum point, and fast evaporation rate increases quickly. This finding may cause the degradation of ingredients that can reduce the yield obtained and damage the composition of essential oil contents [25].

High microwave power will accelerate the heating of the extracted material because linearly, with high power, the higher the heating temperature of the extracted material. High power conveys thermal energy to the material contacted and influences the temperature profile. The oil glands in lemongrass get overheated, breaking the cell walls and lysis process. Oil will diffuse to the solvent and evaporate with the solvent vapor, which is then condensed. High microwave power does not guarantee a high yield because each material has distinct characteristics. Therefore, the best operating conditions are needed to produce high yields and good-quality citronella oil. Based on the experiments that have been carried out in the manufacture of essential oil can be carried out at optimum power for the extraction of citronella oil by the MHD method based on Fig. 2 is 450 W [26].

Effect of Material Size of % Yield of Citronella Oil

Material size variations in the extraction process are 0.5, 1.0, and 1.5 cm long. This size affects the surface area of the material submerged in the solvent. In addition, it also affects the number of material matrices included in one experiment. The effect of material size on yield can be seen in Fig. 3. Fig. 3 shows it can be seen that the yield of citronella oil for a size of 0.5 cm (1.572%) has the highest yield when compared to other sizes, 1.0 cm (0.976%) and 1.5 cm (1.272%). Also, Fig. 3 shows a decrease in the yield of citronella oil as the length of the lemongrass leaves increases. This fact is because the longer the piece of lemongrass, the fewer oil glands are exposed to heat, thereby reducing the surface area of the extracted material so that the smaller the pieces of lemongrass leaves, the more yield is produced. Based on the experiments that have been carried out, optimization in the manufacture of essential oil from lemongrass can be done using a piece of material size of 0.5 cm [27].

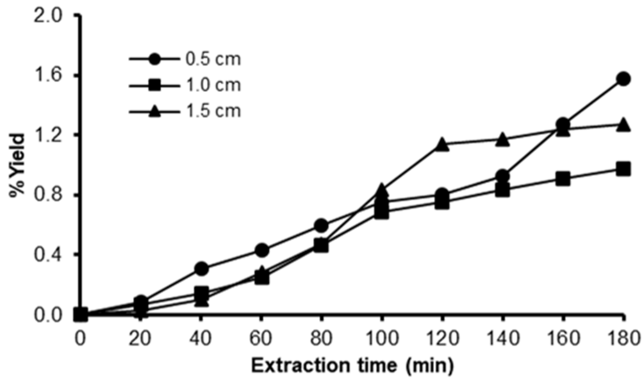

Fig 3. The oil yield function of time for different material sizes

According to the literature, the smaller the size of the extracted material can cause penetration from microwaves to be more effective. Where with the increasingly effective penetration of microwaves in smaller-sized materials, this then causes the extraction efficiency to increase. Based on Fig. 3, it can be seen that the yield of citronella oil for a size of 0.5 cm (1.572%) has the highest yield when compared to other sizes, 1.0 cm (0.976%) and 1.5 cm (1.272%). Where with the increasingly effective penetration of microwaves in smaller-sized materials, this then causes the extraction efficiency to increase [28].

Effect of Feed to the Solvent Ratio of Yield of Citronella Oil

This research was conducted at the ratio of raw materials used per volume of solvents was 0.1, 0.15, 0.2 g/mL, and the mass of material used is 30, 45, and 90 g.

Increasing feed to solvent in the extraction flask causes a yield decrease. It caused by the surface of the material is not exposed to heat optimally. The flask becomes dense, and the heat distribution becomes not optimally spread. The dense flask resists heat transfer between materials so that it decreases extraction energy thermal. Based on Fig. 4, it is found that the optimal yield in the MHD method for citronella oil can be done using the best ratio, where the optimum ratio for the manufacture of citronella oil is about 0.1 g/mL [29].

The volume of the solvent remains constant while the mass of the material changes. It was found that increasing the solid mass can reduce the surface area

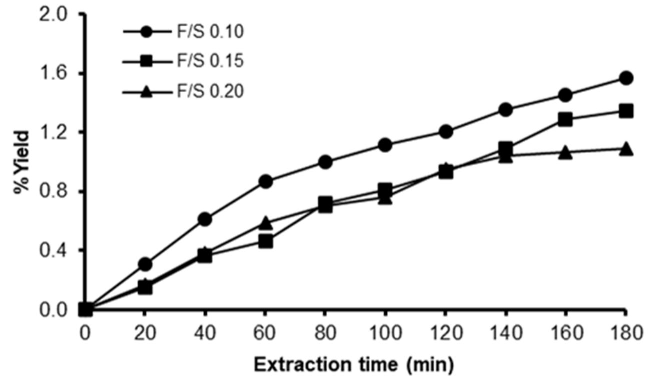

Fig 4. The oil yield function of time for different of feed to solvent ratios (F/S)

available for the solvent to penetrate the plant material and dissolve the target molecules. In general, a lower material-to-solvent (F/S) ratio in extraction techniques can increase the yield of essential oils. The solvent volume should be sufficient to ensure the entire sample is ideally submerged so that the material can expand during extraction [30].

The interaction between power parameters, time, comparison of feeds and solvents, and material size will be discussed in a separate study of optimization and interaction between parameters.

Effect of NaCl Salt Addition on the Yield of Citronella Oil

The best variable for citronella is a material size of 0.5 cm, F/S 0.1, with a material mass of 30 g with a power variation of 300, 450, and 600 W. This research uses NaCl salt to make NaCl 2% solution for the experiment. According to this research, the yield of essential oil increases at NaCl 1–2% and decreases at NaCl concentrations of 3–5% [31].

Another research stated that the yield of essential oil increases in NaCl by 1% and 2.5% but will experience a decrease in yield at NaCl concentrations by 5 and 10%. So from the two research above, variables were selected for extracting clove stem essential oil by the MHD method using NaCl solution 2% [32].

The addition of NaCl accelerates the phenomenon of mass and heat transfer because the addition of NaCl causes the boiling point of water to increase, which can result in the degradation of components, and a hydrolysis reaction occurs [33], so the extraction process can run faster. NaCl act as an electrolyte where NaCl solution consisting of ion moves and rubs against each other due to the influence of electromagnetic waves on the microwave so that the separation of essential oil from water becomes easier [34]. NaCl increases the polarity of the solvent to separate oil from solvent easier.

The selection of solvent is one of the crucial factors that can significantly influence the extraction process using the microwave. The determination of the solvent depends on the solubility of the essential oil, the penetrating power of the solvent, the interaction between the solvent and the matrix of the material, and the dielectric constant [35]. This research used Water and NaCl solutions as solvents because the highest dielectric constant among all solvents is 80.4 and 78 [36]. This fact causes the solvent to have a high capacity to absorb microwaves, and essential oil in lemongrass leaves can be extracted optimally. In this experiment, we used water and added a NaCl solution of 2%.

Fig. 5 shows, on the use of a 2% NaCl solvent, it can be seen that yield increases as the power increases, but there is a decrease in the immense power; yield increases from the power of 300 W (1.094%), 450 W (1.395%) as the power used increases. However, at a power variation of 600 W, there was a decrease (0.988%).

From Table 1, it can be seen that the yield of citronella essential oil has changed at a solvent ratio of 2% NaCl, namely for variables of 300 W (1.394 to 1.094%), 450 W (2.110 to 1.395%), and 600 W (0.904 to 0.988%). The addition of NaCl can affect the dielectric constant to get lower, resulting in the solvent not quickly capturing waves from the microwave so that heating is reduced, this is called dielectric decrement, but this does not happen because dielectric decrement occurs in molarity above 1.5 M. The molarity of the NaCl used is still below that number, so there is no dielectric decrement [37]. This result follows research conducted by Perez et al., where there was a decrease in oil yield from 0% NaCl to 1% NaCl and an increase after adding 2.5% NaCl [32]. The increase in yield, along with the increase in the solvent ratio, is due to the increase in the boiling point of water.

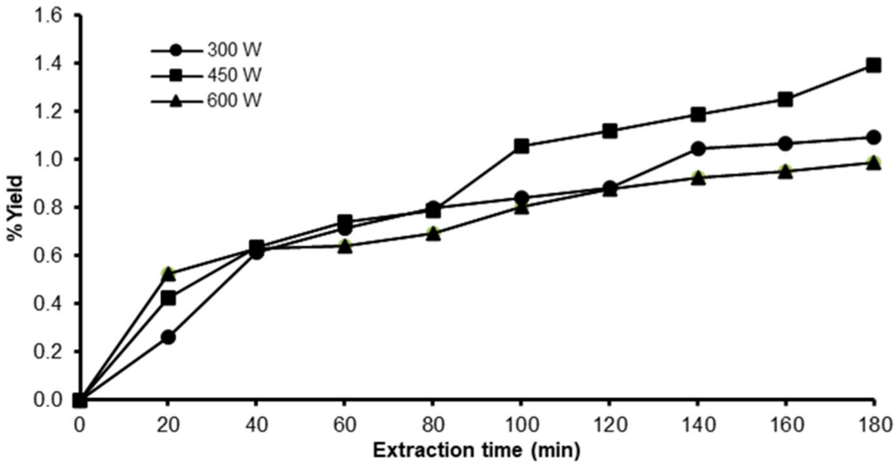

Fig 5. The oil yield function of time for different addition of NaCl 2% solution

Table 1. Comparison of the oil yield between water solvent and 2% NaCl solution

Power	Ratio F/S	Material size (cm)	% Yield (Water)	% Yield (NaCl 2%)
300			1.394	1.094
450	0.1	0.5	2.110	1.395
600			0.904	0.988

Physical Properties of Citronella Oil

The analysis results of the physical properties of citronella oil can be seen in Table 2. The specific gravity of citronella oil processed using the MHD method is 0.896–0.915 g/mL and is close to SNI 06-3953-1995, which is 0.88–0.92 g/mL, meaning essential oil has met quality standards in Indonesia. The specific gravity of citronella oil extracted in this research is almost close to Putri's research [38], whose specific gravity is 0.8640–0.9087. The greater the weight fraction contained in the oil, the greater the density value. The greater the density value, the more components are contained in the substance with a high molecular weight and a long carbon chain [39].

Essential oil dissolves in ethanol 70% at a specific ratio and concentration. The solubility in ethanol of 70% states the ratio of the volume of essential oil and the volume of ethanol of 70% needed to dissolve essential oils. The solubility test on citronella oil obtained the results that the solubility of the citronella oil obtained was following the SNI standard, which was 1:2. This is also reinforced by the results of experiments conducted by Putri [40] that citronella oil dissolves in 70% ethanol in a ratio of 1:2, that is, 1 mL of citronella essential oil is required 2 mL of ethanol, so a clear solution is obtained. The solubility test in alcohol gives an idea of whether an oil is easily soluble or not. The easier the oil is soluble in alcohol, the more polar compounds in the oil. The solubility of alcohol is a principal factor in essential oil testing because it can determine the quality of the essential oil.

The refractive index of citronella oil is 1468, which means that the value of the refractive index of the extracted oil is under the SNI standard, 1466–1475. The refractive index of essential oils is closely related to the

Table 2. Physical properties of citronella oil

Physical properties	SNI 06-3953-1995	Citronella oil
Specific gravity (25 °C)	0.88–0.92	0.896–0.915
Solubility 70% (v/v)	1:2	1:2
Refractive index	1466–1475	1468
Color	Pale yellow to yellowish brown	Pale yellow to yellowish brown

components composed in the resulting essential oil. Similarly, the specific gravity of the essential oil constituent components can affect its refractive index value. The more long-chain components such as sesquiterpenes or oxygen components are distilled, the density of the essential oil medium will increase, making the incoming light more challenging to refract. This finding leads to a more extensive oil refractive index. According to Guenther and Ketaren [39], the index value is also influenced, one of which is the presence of water in the oil content. The bias index value decreases as the water content increases. This fact is due to the nature of the water that it is easy to refract the oncoming light.

Results of Analysis Chemical Properties of Citronella Oil

The results of the analysis of the contents of citronella oil are provided in Table 3, and the chromatogram is displayed in Fig. 6. The predominant component of the essential oil extracted from lemongrass using the MHD method is geraniol, as shown in Fig. 6.

Citronelal and geraniol experimental results for the MHD method were 5.62 and 46.61%. In a previous study by Putri, the main components of citronella oil from lemongrass leaves extraction by the MHD method are geraniol and citronellal 28.44 and 6.85%, respectively. This result does not correspond to the expected essential oil, where the components of geraniol and citronellal,

Table 3. Result of GC-MS analysis of citronella oil

Retention time (min)	% area	Compounds
1.723	0.70	3-Methyl pentane
1.767	23.41	Hexane
1.892	1.80	Methyl cyclopentane
7.873	0.36	Linalool
8.693	3.00	Citronella
8.864	0.37	Isoneral
9.136	0.67	Isocitral
9.822	2.62	Citronella
10.044	10.35	Geraniol
10.226	19.65	Geraniol
10.469	16.61	Geraniol
11.545	0.50	Butanoic acid
11.707	9.71	Eugenol
11.955	3.21	Geranyl acetate
12.610	1.69	Caryophyllene
12.933	0.83	Phenol
13.390	0.47	Germacrene
13.785	0.98	Naphthalene
14.565	0.84	Cyclodecadiena
14.685	0.95	Caryophyllene
15.327	0.52	Naphthalene
15.481	0.76	Cyclohexane
Total	100	

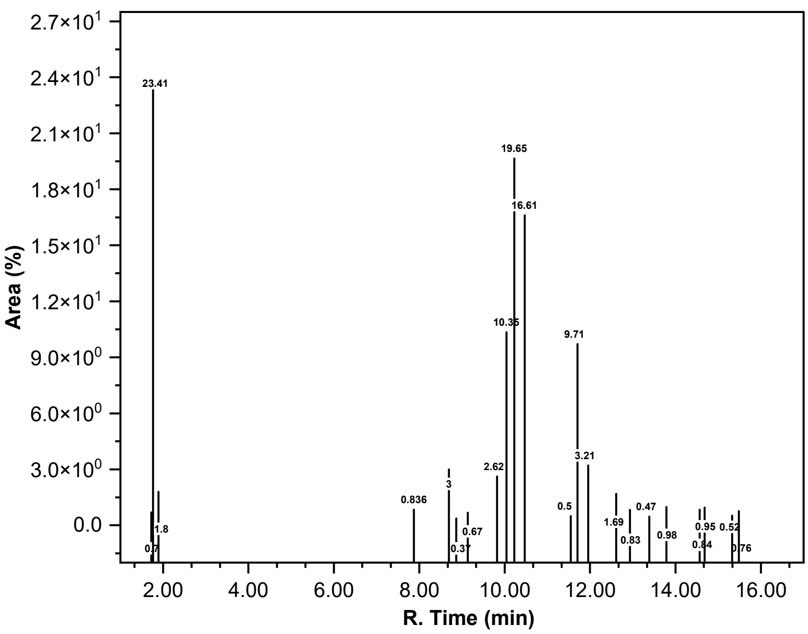

Fig 6. Gas chromatography-mass spectrometry chromatogram

according to SNI. 06-3953-1995, are respectively 85 and 35%. According to Widyastuti and Sugiarso [41], genetic and environmental factors cause variation in the levels of geraniol and citronellal in this study. The availability of nutrients in the soil results in differences in the results of plant metabolic processes [42].

■ CONCLUSION

The extraction of lemongrass leaves by the MHD Method shows that the smaller the material size affects, the greater the yield with an optimum size of 0.5 cm. The best value of the power of the MHD method's extraction process of lemongrass leaves is 450 W. As the ratio of solvents used increases, the yield obtained increases. The best solvent per material ratio value is F/S = 0.1, and the best solvent for the extraction process of lemongrass leaves by the MHD method is aquadest. Physical and chemical results of the analysis of citronella oil analyzed by GC-MS show that citronellal and geraniol components get results of 5.62 and 46.61%, respectively. The solubility analysis of citronella oil in alcohol is 70%, following previous research and Indonesian National Standardization results. The yield of citronella oil obtained from extraction by the MHD method using water solvents is more remarkable than using 2% NaCl, and this is because the dielectric constant of NaCl is more diminutive than water and needs to be studied further for higher NaCl concentrations.

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