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EXTRACTING ESSENTIAL OIL FROM FRAGRANT LEMONGRASS PLANTS BY STEAM-HYDRO DISTILLATION METHOD USING MICROWAVE AS A HEATER

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ABSTRACT

This research studies the process of extracting citronella oil by the steam-hydro distillation method with microwaves (microwave). In addition, study the effect of temperature (105 ° C, 110 ° C, 115 ° C) and the size of the material enumeration (0.5 cm; 1 cm; 1.5 cm) on the yield of oil produced. This research was conducted by the steam-hydro distillation method. In this method a solvent is added in the form of water to dissolve the oil in the fragrant lemongrass stems and leaves. The resulting steam is condensed and then distillate in the form of a mixture of oil and water is taken after 120 minutes and separated using a separating funnel. From the results of the study, it was found that the% yield of high citronella oil in the variable size of the enumeration material was 0.5 cm at a temperature of 115 with a yield of 1.92%. The content of Citronella obtained in the oil of this research is 59.28%. The density of lemongrass oil is 0.875. The refractive index value is obtained in the range of 1.46735- 1.46925.

Keywords : *Essential Oil, Fragrant, Steam-hydro Distilation, Microwave*

1. Introduction

Along with the development of modern industries such as perfume, cosmetics, food, aroma therapy and medicines industries, the need for essential oils in the world is increasing. Essential oils have now been developed and become Indonesian export commodities which include essential oils from patchouli, fragrant roots, nutmeg, cloves, citronella, cananga, eucalyptus, sandalwood, pepper, and cinnamon (Feriyanto, 2013). Fragrant lemongrass is one of the most prospective essential commodities and is rich in benefits. Demand for citronella oil is quite high and the price is stable and tends to increase so that the citronella oil processing industry contributes greatly to improving the economy of the community.

The main problem in the citronella oil refining industry is the refining capacity that is not able to meet consumer demand, due to the working methods and refining machine quality that is not yet sufficient to produce citronella oil with maximum quality and quantity, therefore efforts are needed to improve industrial performance distillation of essential oils, by means of utilizing appropriate refining technology to increase refining capacity and increase the amount and quality of essential oils produced (Nugraha, 2017). Essential oil can be obtained through several separation methods including hydrodistillation which is the most conventional practice (Farahnaky, 2018). Different separation methods such as distillation (hydro and steam), solvent extraction and supercritical fluid extraction can be used to extract essences or volatiles. However, the quality and quantity of oil yields depend on the extraction technique used (Chanthai,

2012). The essence or volatile obtained by solvent extraction can contain traces of solvents which may have damaging effects. On the other hand, supercritical fluid extraction is an efficient technique for producing high yields and essences that are of good quality or volatile, but the initial cost of investment is very high. Therefore, a relatively simple, inexpensive, environmentally friendly distillation method, producing good quality oils is generally preferred for extracting essential oils from citronella (Manaf, 2013). To get essential oils from citronella requires a relatively long time, which is about 4-7 hours using hydro distillation and steam distillation (Feriyanto, 2013).

In order to make the extraction time shorter and the resulting yields of higher quality and increased, the extraction of essential oils from lemongrass stems and leaves is improved by using technology, so that microwave is used as a new method to achieve the target. The time required for a microwave is relatively shorter to obtain the same yield for the hydro distillation and steam distillation methods because the microwave is more effective and efficient in heat distribution. Based on that, it is necessary to study the distillation of the fragrant lemongrass stems and leaves with a modification method of the study of steam and hydro distillation with the help of microwave as a heater.

Essential Oil of Citronella Oil

Essential oils are the essence of aromatic plant species obtained by hydrodistilation or steam distillation of whole plants or from certain parts such as flowers, fruit, leaves, roots, bark and seeds (Mu'azu, 2012). Essential oils can be obtained from patchouli, fragrant roots, nutmeg, cloves, lemongrass, cananga, eucalyptus, sandalwood, pepper, and cinnamon which includes the leaves, flowers, stems and roots (Richards, 1944). Fragrant lemongrass (Cymbopogon Winterianus Jowitt) is a plant in the form of upright grasses, and has very deep and strong roots, upright stems, forming clumps. This plant can grow to a height of 1 to 1.5 meters. The leaves are single leaves, complete and the leaf stem is cylindrical, bare, often the inner surface is red, the tip is tongued, with a length of up to 70-80 cm and a width of 2-5 cm (Segawa, 2007).

Fragrant lemongrass plants (Cymbopogon Winterianus Jowitt) can live in areas where the air is hot or cold, up to a height of 1,200 meters above sea level. How to breed with children or shoots roots. This plant can be harvested after 4-8 months. Harvesting is usually done by cutting the clump near the ground (Soebardjo, 2010). Flower arrangement of fragrant lemongrass branches, stemmed, usually the same color and generally white. Fragrant lemongrass rarely blooms and only blooms when it is ripe enough, ie at the age of more than 8 months. The petals metamorphose into 2 lodic glands, functioning to open flowers in the morning. Stamen amounted to 3-6, a pair of book-shaped pistil heads with a crested-shaped extension (Segawa, 2007).

In Indonesia, lemongrass plants can generally be classified into two groups, namely: lemongrass or bamboo lemongrass (Cymbopogon Citratus) and citronella or citronella citronella (Cymbopogon Nardus). Generally we do not distinguish between the names of fragrant lemongrass and lemon lemongrass, although both types are easily distinguished (Harris in Ginting, 2004). In terms of its chemical components, citronella oil and kitchen citronella oil have different main components. Lemongrass is the main content of citronellal, while the lemongrass is citral.

Cymbopogon Winterianus Jowwit, "expert" (Andropogon Nardus Java de Jong). Mahapengiri has the characteristics of broader leaves and shorter, besides that it produces oil with high levels of citronellal and geraniol. The yield of oil

produced from lemongrass leaves depends on a variety of factors, including: climate, soil fertility, plant age and ways of refining. According to De Jong the yield of oil from fresh leaves is around 0.5-1.2% and the yield of oil in the dry season is higher than in the rainy season (Ketaren in Utomo, 2009).

Plant Classification

The classification of fragrant lemongrass plants is as follows:

Kingdom	: Plantae (Plants)
Divisio	: Spermatophyta (Plants produce flowers)
Sub Division	: Angiosperms (Covered seed plants)
Class	: Monocotyledoneae (One-Piece Plants)
Marga	: Cymbopogon
Type	: Cymbopogon Winterianus Jowitt ex Bor

Uses Lemongrass Fragrance

Lemongrass has a property as a sinusitis medication or respiratory problems. The essential oil extract can be used as a liniment. Lemongrass stems can be boiled in warm water and used as a fragrance in the bath tub, the benefits are to refresh the body and relax tense muscles. The oil produced from citronella extract can be used to repel mosquitoes and protect from mosquito bites. Fragrant lemongrass (Cymbopogon Winterianus Jowitt) as a traditional medicinal plant, its roots have efficacy as a urine drop, sweat drop, phlegm drop (cough medicine), mouthwash, and body warmers. The leaves as a cold medicine, appetite enhancer, postpartum treatment, fever reducing and seizure relief (Wibisono, 2011).

Lemongrass is widely used in Malay, Indonesian and Thai cuisine. In addition to the leaves, lemongrass oil can also be taken that can be used as a deodorant bath soap or perfume, which we know better as fragrance oil. The parts of the lemongrass plants that are useful and useful, namely: washing the fishy odor in meat, lemon grass boiled water can be used for bath water, roots and stems treat stomach pain, help balance hormones. Citronella oil is an essential oil which is known for its natural insect repellent properties and is very attractive to the pharmaceutical and fragrance industry (Wany, 2013). In traditional practice, citronella oil has been used as an antipyretic, aromatic, vermifuge, diuretic and mental illness tea (Wany, 2014).

Chemical Composition of Fragrant Lemongrass Oil

Essential oils from fragrant lemongrass obtained by distillation from the leaves and stems of fresh lemongrass by steam distillation method with essential oil content of 0.5-1.2% (Ginting, 2004). The main ingredients of essential oils are citronellal, citronellol, geraniol, and citral. The amount of compound content is also related to the plant species. Cymbopogon Winterianus Jowitt has the highest content of citronellal and geraniol (Arswendiyumna, 2006).

The main chemical composition of fragrant lemongrass oil is monoterpenes, alcohol and aldehydes, so that essential oils have physical and chemical properties that are included in the alcohol class. Geraniol is a compound consisting of two isopropene molecules, whereas citronellol is the result of condensation from citronellal included in the aldehyde group. With oil content like this, the evaporation is included in the group of fast to moderate (top to middle note). Citronellal and sititral content have potential biological effects as analgesics, which provide a calming effect and pain reduction (De Sousa and Damio, 2011). The chemical components in citronella oil are quite complex, but the most important components are geraniol and citronellal. These two components determine the intensity of odor, fragrance, and the value of citronella oil price. The content of the main components of fragrant lemongrass oil is not fixed, and depends on several factors. Geraniol levels

are usually high then the citronellal levels are also high (Harris in Ginting, 2004). Composition of citronella oil consists of several components, some have 30-40 components, the contents of which include alcohol, hydrocarbons, esters, aldehydes, ketones, lactones, terpenes, and so on.

Table 1. Chemical Composition of Citronella Oil

Compounding Compounds	**Percent (%)**
Sitronellal	Min. 35
Geraniol	12 – 18
Sitronellol	12 – 15
Geraniol Asetat	3 – 8
Sitronelil Asetat	2 – 4
1 - *Limonene*	2 – 55
Elemol & *Seskwiterpene* lain – *Elemmene* dan – *Cadinene Seskwiterpene* yang terdiri dari : *Eugenol, Metal Eupenol, Isopulegol, Nerol, Linalool, Sitral, Metal Heptenon, Myrcene dan Pinene*	2 – 5

(Sumber: Ketaren, 1985)

The main components of fragrant lemongrass oil are as follows:

✓ Geraniol (C10 H18 O)

Geraniol is the main constituent of lemongrass oil. Insoluble in water and soluble in organic solvents. Geraniol is a compound consisting of 2 isoprene molecules and 1 water molecule, with the following formula:

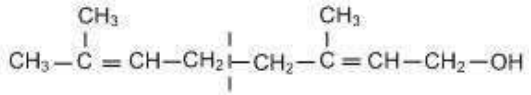


Figure 1. The Geraniol Build Formula

✓ Sitronellol (C_{10} H_{20} O)

Citronellol is found in rose oil and lemongrass oil. At room temperature the liquid form is colorless and smells rose, can dissolve in alcohol and ether, but slightly soluble in water. Have a formula like this:

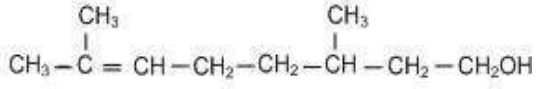


Figure 2. Sitronellol Build Formula

✓ Sitronellal (C_{10} H_{16} O)

Citronellal compounds are found in citronella oil, lemon grass, and roses. At room temperature, the citronellal liquid is yellowish and volatile, is slightly soluble in water and soluble in alcohol and esters. Smells pleasant and is used for perfume in soap. Have a formula like this:

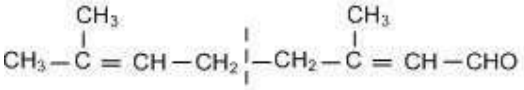


Figure 3. Sitronellal Build Formula

Quality Standards for Citronella Oil

The main cause of pleasant odors in citronella oil is citronellal which is the basic ingredient for perfume, and therefore citronellal oil with high citronellal levels is more popular. Such oil will be obtained from the first refining fraction. Specifically in Indonesia, traded citronella oil is obtained by distillation of the leaves of the Cymbopogon Nardus plant. Indonesian citronella oil is classified in one main quality type by the name "Java Citronella Oil" (Ketaren in Utomo, 2009). The quality standard of citronella oil for export quality can be analyzed according to physical criteria, namely based on: color, specific gravity, refractive index, or chemically based on total geraniol and total citronellal (Kapoor and Krishan in Utomo, 2009).

Table 2 Quality Standards for Fragrant Lemongrass Oil in Indonesia

Characteristics	**Requirements**
Colour	Pale Yellow - Brownish Yellow
Specific Weight 25°C	0.850 – 0.892
Bias Index 25°C	1.466 – 1.475
Total Geraniol	Min. 85%
Sitronellal	Min. 35%

Solubility in ethanol 80%	1 : 2 Clear, so clear
Fat	Negatif
Additional Alcohol	Negatif
Pelican oil	Negatif

(Source: Ketaren, 1985)

Parameters of Essential Oils

Some parameters that are usually used as standards to recognize the quality of essential oils include:

- ✓ Specific Weight

The value of essential oils is defined as the ratio between the weight of oil and the weight of water in the same volume of water as the volume of oil. (Sastrohamidjojo, 2004).

- ✓ Bias Index

Refractive index is the ratio between the speed of light in the air with the speed of light in the substance at a certain temperature. The refractive index of essential oils is closely related to the components arranged in the essential oil produced. (Sastrohamidjojo, 2004).

- ✓ *Gas Chromatography Mass Spectrometry (*GC-MS)

This analysis uses the combining of 2 tools namely Gas Chromatography (GC) which functions to analyze the molecular structure of compounds and separate chemical fractions in compounds and Mass Spectrometry (MS) which functions to analyze the quantity of compounds quantitatively (looking for chemical contents in compounds and mass of particles and concentration). Gas Chromatography is a separation technique based on differences in the migration speed of the constituent components.

Distillation

Distillation is the separation of two or more components based on differences in the boiling point between compounds and the boiling point of the separated compound must be much different. Distillation commonly used there are 3, such as:

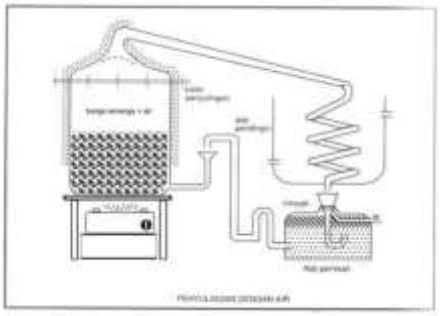


Figure 4. Hydro Distillation

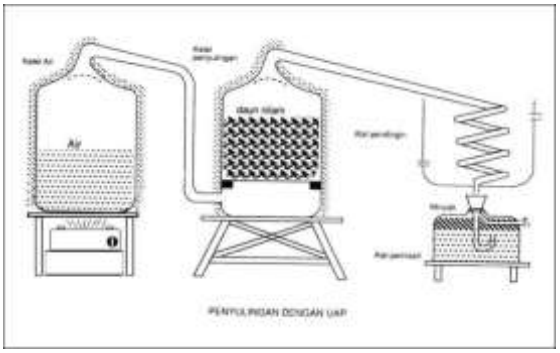


Figure 5. Steam-Hydro Distillation

- ❖ Air Water distillation (Hydro) is distillation that uses a solvent (usually water) on a material, so the material is immersed and a distillation process is carried out.
- ❖ Steam distillation is distillation that uses the addition of steam to the material to be distilled. Material is not immersed in a solvent.
- ❖ Steam and Water Distillation (Steam and Hydro) is a mixture of the two processes above, where the material is soaked with solvent and at the same time steam is added.

Microwave (Oven)

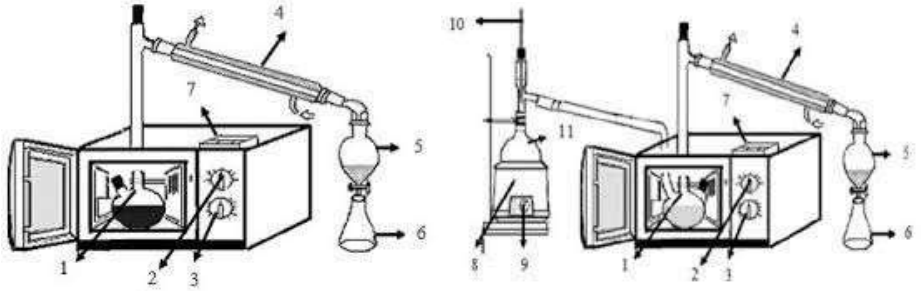
Microwave (micro wave) is one of the electromagnetic waves which has a frequency of 2.45 GHz with a wavelength of 1 mm - 1 m. Microwave is a shortwave that is very useful from the beginning of the development of radar, missiles, television and others. The microwaves that are around us are found in ovens for heating food and this is called a microwave oven. Microwave ovens consist of several components such as:

Transformer / transformer, Magnetron, waveguide, stirrer, and metal wall.

Working Principle of Microwave

Microwave emitted from a magnetron radiates through the waveguide and into the food, the food is rotated by a stirrer so that heating is more evenly distributed. Microwave is very sophisticated, which reflects waves that hit glass and plastic so that the waves will only hit the target, which is food. Microwave heating occurs in all parts of the food and is different from heating with a regular stove, the heat starts from the outside just inside, so when the inside begins to heat the outside is charred. The principle of microwave heating is that the transmitted waves are absorbed by the water molecules present in the food, because they are exposed to energy and the water molecules will move randomly, causing continuous collisions, rubbing and heat. So the food will be cooked throughout.

Figure 6. Microwave



(a) (b)

Keterangan :

1. Labu Leher Dua
2. Pengatur Suhu
3. Pengatur Timer
4. Kondensor
5. Corong Pemisah
6. Erlenmeyer
7. Termokopel
8. Heating Mantle
9. Pengatur Suhu
10. Thermometer
11. Labu

Figure 7. (a) Schematic of Hydro Distillation with Microwave Heating, *(b)* Schematic of a Hydro-Steam Distillation device with Microwave Heating

2. METHOD

This research was conducted at the Phuga Aceh Foundation Lhokseumawe Research, Aceh and the study was conducted in March 2019. Tools and materials used were Microwave, Pumpkin neck three, Pumpkin neck two, Heating mantle. Water hose, Condensor, Separator

Funnel, Thermometer, Refractometer, Pycnometer, GC-MS. while the ingredients used are fragrant lemongrass plants and water

Raw Material Preparation:

Fragrant lemongrass plants were obtained from the Simpang Kramat area from community agriculture

Experiment Procedure

Fragrant lemongrass leaves and stems weighed 100 grams each, lemongrass leaves and stems that had been weighed were chopped with a size of 1.5 cm; 1 cm; 0.5 cm, the scaled and chopped lemongrass leaves and stems are put into a three neck distillation flask, and 500 mL of water is added as a solvent. Heated water in a two neck flask using a heating mantle for use as a steam generator, then turned on the microwave heater and adjusted the temperature according to the variations used, and set the timer rotation for 120 minutes. Distillation time is calculated starting from the first drop out of the condenser. The process is stopped after 120 minutes then the distillate is accommodated in a separating funnel, and oil is separated from the water and oil is taken free of the water content for analysis of the resulting oil.

Prosedur Uji
Uji GC – MS

Component analysis is carried out by means of GC-MS from the yield of oil produced can be carried out according to the method of quality test for citronella oil listed according to the standard reference (SNI 06-3953-1995).

Bias Index Test

Refractive index analysis is carried out with a refractometer from the yield of oil produced can be carried out according to the method of quality test for citronella oil listed according to the standard reference (SNI 06-3953-1995).

3. RESULT AND DISCUSSION

Hydrodistillation is the method most widely used in the extraction of essential oils (essential oils), one of which is citronella, because it has many advantages, such as: easy operation, and an environmentally friendly process. In this hydrodistillation method using Microwave as a heater, because to get essential oil (essential oils) from citronella fragrance usually requires a relatively long time which is about 4-7 hours so that technology is improved by using a microwave so that the uptake time is shorter and the yield produced higher quality and improved due to microwave waves absorbed from water molecules and cause an increase in water temperature, both solvent water and water contained in the material quickly. Increasing the temperature of the water in the material can lead to rupture of the material and increase the oil diffusivity.

Figure 8. Range of equipment

From the research conducted, obtained a yield in the form of essential oils (essential oils) lemongrass fragrance that has the following characteristics: pale yellow, has a distinctive aroma of lemongrass and the texture of oil that is not too thick. The yield is obtained through the hydrodistillation method which is used as a microwave heater. Before the hydrodistilation process is carried out, fragrant lemongrass which is still in the form of leaves and stems is

chopped in advance with a variety of enumeration sizes, namely 0.5 cm; 1 cm; and 1.5 cm. The purpose of enumeration is to expand the evaporation area and the contact surface between the material and water so that it is more easily extracted. After the chopped lemongrass was obtained, the chopped was weighed as much as 100 grams and put into a feed flask for hydrodistilation which in this method used water as a solvent. The ratio between ingredients and solvents is 1: 5, so the water used is 500 mL. The temperature for the hydrodistillation process is also varied for each variation of the size of the enumeration ie for each size of the enumeration is varied again with a temperature of 105 C; 110 ° C; and 115 ° C. The purpose of varying temperature is to see how the influence of temperature on the yield of oil produced. To further accelerate the heat, a steam generator in the form of water that is heated in a heating mantle whose temperature is set to 100 ° C, then the resulting steam is flowed into the feed flask, so that steam from the flask can more quickly follow the steam from the heating mantle. Therefore, this method is also called the steam-distillation method, because it uses steam to accelerate the heating process. The results of this study in the form of essential oil (essential oil) with the most yield yielded at variations in the size of the enumeration of 0.5 cm at a temperature of 115 yaitu C, which is as much as 1.92% and the least is the variation of the size of the enumeration of 2 cm at a temperature of 105 ° C namely as much as 0.61%, from the results of the study it can be seen that the smaller the variation of the size of the material carried out, the greater the yield of oil produced due to the wider contact surface between the material and the solvent (water).

The results of examination with GC-MS, showed that this material contains 63 components, according to the results of GC-MS examination this material contains 59.28% Sitronellal, 9.57% Sitronellol, and 20.32% Geraniol. According to international market standards, Sitronellal content must be higher than 35%. Therefore this material meets the quality standards of the international market and besides the physical and chemical properties of citronella oil used as research material it also meets the quality requirements based on the Indonesian National Standard (SNI).

Figure 9. Mixture of water + oil that enters the distillation flask

The following are the results of the GC-MS examination on citronella oil research results:

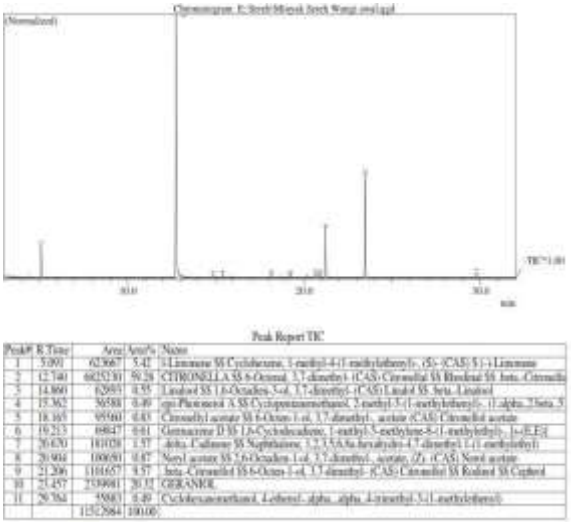


Figure 10. GC-MS Test Results of Fragrant Lemongrass Oil

The sequential number of the components above shows the order in which the "peak" image came out from the relevant components, when analyzed using GC-MS. This actually can also be used as an indication to find out the amount of boiling point of the component in question. The more recent peak release of GC-MS analysis results from a component means the higher the boiling point of the component concerned, the more recent release of the peak GC-MS analysis results from a component means the higher the boiling point of the component concerned

a. Bias Index

Refractive index describes the spread of light through the media measured by a refractometer. This is also used to distinguish water from other solvents as well as to determine the purity level of essential oils. Oil quality can be seen from its refractive index. Essential oils with large refractive index values are better than essential oils that have small refractive indices.

Table 3**.** Research result :

Ukuran Pencacahan (cm)	**Suhu (^oC)**	**Volume (mL)**	**Rendemen (%)**	**Indeks Bias**
0,5	**105**	1,7	1,48	1,4692
	110	2	1,74	1,4690
	115	2,2	1,92	1,4686
1	**105**	1	0,87	1,4674
	110	1,7	1,48	1,4673
	115	2	1,74	1,4690
1,5	**105**	0,7	0,61	1,4686
	110	1,5	1,31	1,4690
	115	1,8	1,57	1,4685

Determination of the refractive index is done by refractometer at room temperature. Furthermore, the refractive index calculation is in accordance with the Indonesian National Standard, which is at a temperature of 20 ^{o} C. Based on SNI 06-3953-1995 for good quality citronella oil, the index of citronella oil at a temperature of 20 ^{o} C is in the range of 1.466-1.475. In the following figure, it can be observed the results of the refractive index analysis of the range based on SNI.

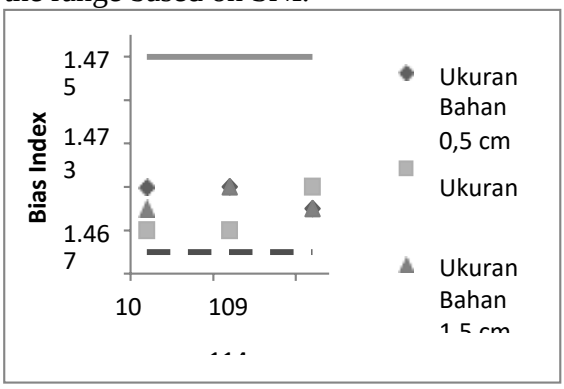


Figure 11. Refined Citronella Oil Bias Index Against Quality Standards (SNI)

From Figure 9, it can be seen that all parameters are within the refractive index range based on SNI. The refractive index is closely related to the content of the components arranged in the citronella oil produced. From Figure 9, it is found that the small amount of water contained in oil does not reduce the quality of the refractive index.

4. CONCLUSION

From the research results the process of extracting essential oils from fragrant lemongrass plants can be concluded that: The effect of the operating temperature

which results in a high% yield is the highest operating temperature of 115°C, and the effect of the enumeration size that results in a high% yield is when the smallest enumeration size condition is 0.5 cm, while the refractive index value of the citronella oil is The results are in the range 1.4673 - 1.5692, these results have met the quality standard (SNI). The resulting oil density was 0.875 gr / mL, according to GC-MS results, the citronella oil produced contained 3 main components, each of which was as follows:
Sitronellal = 59,28 % Sitronellol = 9,57 % Geraniol = 20,32 %

These results meet the quality standard (SNI). In taking essential oils from citronella plants using the Hydro-Steam Distillation method with Microwave heating, the highest yield was 1.92%.

5. ACKNOWLEDGEMENT

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