

REFINERY OF CITRONELLA OIL USING VACUUM HYDRODISTILLATION METHOD IN ACEH UTARA DISTRICT

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ABSTRACT

Citronella oil is a non-timber forest product (NTFP) and is commercially obtained from the distillation of citronella leaves (*Cymbopogon nardus* L). Fragrant citronella leaves are one of the raw materials for obtaining vegetable oils in the form of essential oils. Essential oil is the main component consisting of composition in the form of geraniol and citronellal. The process of extracting essential oils can be done using various methods, one of which is the Vacuum Hydrodistillation method. The Vacuum Hydrodistillation method is carried out using a vacuum pump as a pressure guard and the boiling point of the distillation process does not exceed the boiling point temperature of water, which is 1000 C, so that the oil produced is maintained its purity. The vacuum hydrodistillation process of citronella was carried out for 3, 5, and 7 hours with each treatment using random and whole pieces of raw material. The resulting essential oil products were analyzed in the form of yield, density, refractive index and oil composition analysis using GC-MS. The best essential oil yields from citronella were obtained under operating conditions with random samples and distillation time of 5 hours. Where the yield produced is 0.17% with a refractive index of 1.41570 and a density of 0.92735 gr/ml.

Keywords: Essential Oil, Vacuum Hydrodistillation, Fragrant Lemongrass, Geraniol

1. INTRODUCTION

Citronella oil is one of the most prospective oil commodities among the 12 essential oils exported by Indonesia. Market demand for essential oils is very large, especially for citronella oil, ranging from 9-10%. patchouli and vetiver oil which are in a lower position in terms of demand for citronella oil (vetiver oil) (BPS, 2020).[4,5,6] Some areas that produce citronella oil include Nanggroe Aceh Darussalam, West Java and Central Java with production of more than 95% of total production in Indonesia (Directorate General of Plantations, 2013).[7] With a fairly large number of sales of essential oils, the selling price of these oils is still

affordable and very cheap. This is because the quality of the quality requirements does not meet the qualifications. Limited public knowledge regarding the processing of refining citronella oil is the main key cause for the decline in the quality of citronella oil. [8,9] According to a report from the Ministry of Trade (2011) world citronella oil production is approaching 4000 tons and 40% of its sources are supplied by China and Indonesia. The low production of citronella oil can further increase the value of citronella oil with the presence of derivative products from this oil. Various uses of fragrant citronella oil that can be used such as making cosmetic products, food, beverages. The fragrance can last below the boiling point of water so it

doesn't damage the content of the citronella oil itself.[13,14,15]

2. RESEARCH METHODOLOGY

The raw material used is fragrant citronella. While the equipment used is a set of vacuum hydrodistillation tools, refractometers, pycnometers, measuring pipettes, Erlenmeyer and a set of 50 ml titration tools. 25 liters of water is put into the distilled kettle. [16,17,18] 10 kg of prepared citronella leaves with whole size and random pieces are put into the distilled kettle. The pump is turned on to circulate the cooling water, then the heater is turned on until it reaches a temperature of 100°C and then the distillation process is carried out by varying the boiling time. Variation of boiling time is 3.5 and 7 hours. [19,20] During the boiling process, the steam from citronella leaves will be cooled using cooling water. So the oil will come out through the condenser. After the distillation process is complete, the yield of the oil obtained is calculated and the quality of the essential oil is analyzed. [21].

2.1 GAS CHROMATOGRAPHY-MASS SPECTROMETRY (GC-MS) ANALYSIS

Analysis of citronella oil samples was carried out using GC-MS gas chromatography (GC-Shimadzu 2010 with an autosampler and ionization detector. GC-MS was connected to a mass spectrometer (Agilent 5975C) using a DB-1MS capillary column (30 x 0.25 mm 1. D) 0.25 μ m layer thickness). Injector and detector temperatures were set at 250 °C. Oven temperature was programmed at 40 °C for 8 minutes, increased at 3 °C/min to 240 °C and then held for 10 minutes. Helium as carrier gas was set to flow rate of 1.2 mL/min. The sample volume injected was 10 μ L with the aim of qualitatively identifying the presence of compounds present in citronella oil [22]

2.2 OIL YIELD ANALYSIS

Calculate the weight of the citronella to be refined (input), then calculate the final weight of the fragrant citronella oil obtained (output). [23]

$$\text{Yield (\%)} = \frac{\text{Weight of refined oil (output)} \times 100\%}{\text{Weight of distilled citronella (input)}}$$

2.3 DENSITY ANALYSIS

1. Weigh the empty chemical pycnometer
2. Fill the empty pycnometer with essential oil distillate
3. Reduce the mass of the pycnometer which has been filled with yield with a beaker empty
4. Then the mass and volume are obtained

$$\rho = m/v$$

2.4 BIAS INDEX ANALYSIS

1. Drop the sample to be checked for its refractive index in the sample container Refractometer.
2. Cover tightly and allow light to pass through the solution and through the prism. So that the light on the screen in the tool is divided into two.
3. Slide the boundary mark by rotating the adjusting knob, until intersects the point of intersection of two diagonal lines that intersect with each other on screen.
4. Observe and read the refractive index scale indicated by the screen needle through a microscope.
5. Two-color result display that has been set up so as to provide two colors that have a clear and firm color

3 RESULTS AND DISCUSSION

From the results of research on distillation of essential oils from citronella using the vacuum hydrodistillation method as follows:

Table 1. Results of research

Sample Size	Distillation Time (Hours)	Color	Refractive Index (π)	Acid Number	Density (gr/ml)	Yield (%)
Random Pieces	5	Clear	1.41570	4.32	0.92735	0.17
	6	Clear	1.41508	4.49	0.85838	0.29
	7	Clear	1.41493	4.66	0.86406	0.37
Intact	5	Clear	1.41547	4.32	0.83954	0.13
	6	Clear	1.41484	4.88	0.83000	0.22
	7	Clear	1.41464	4.49	0.82947	0.27

Yield

Below is a graph showing the yield levels for samples of the full size and random pieces.

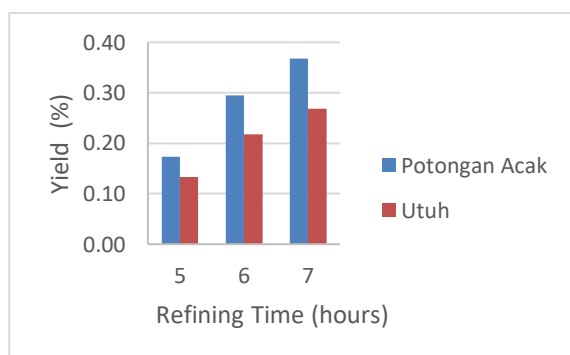


Figure 1.1 Effect of Distillation Time on Essential Oil Yield Levels

From the research that has been done, it is known that the highest oil yield was obtained from the vacuum hydrodistillation process with random sample pieces and 5 hours of distillation time, which is equal to 0.17%. This is because the samples that were randomly cut have a smaller size than the whole pieces the more surface area, so the oil will be easier to extract.

Density

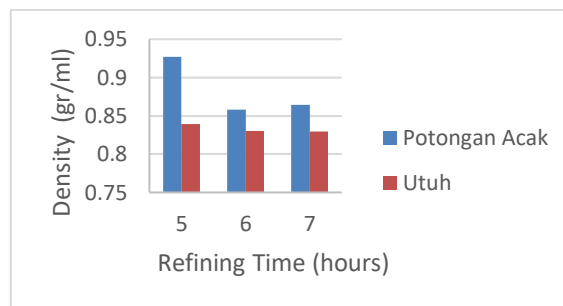


Figure 1.2 The Effect of Distillation Time on the Density of Essential Oils

From the graph above it can be seen that the distillation time affects the density of the essential oil obtained, where the longer the distillation time the lower the density of the oil. And for the best density value, it was found in samples with random pieces and 5 hours of distillation time, which was 0.92735 gram/ml.

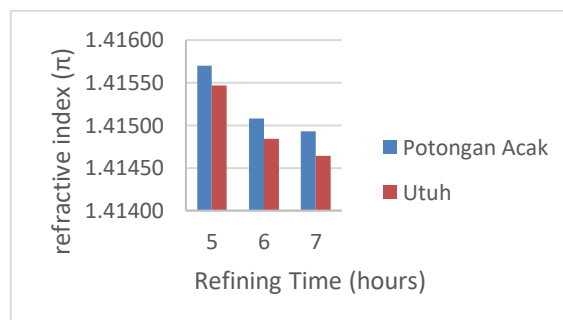
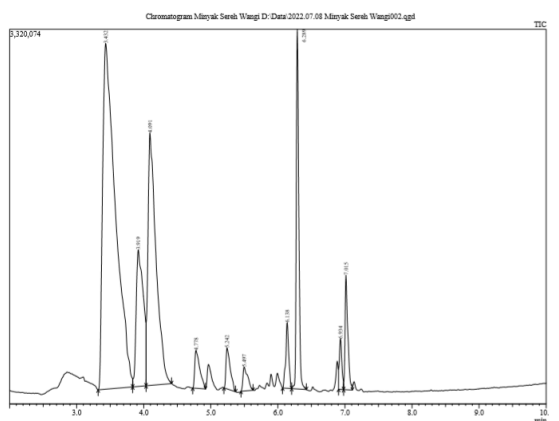


Figure 1.3 The Effect of Distillation Time on the Refractive Index of Essential Oils

From the graph of the relationship between distillation time and the value of the refractive index of the essential oil samples, it can be seen that the distillation time has an effect on the refractive index. Where the longer the refining time, the lower the refractive index. This is because the longer the oil is extracted at a certain temperature, the components of the essential oil will be damaged, so that the value of the refractive index decreases. While the comparison between the size of the intact sample and the random section, it was found that the index of refraction in the

random section was higher than that of the intact sample. According to Ibrahim (2021) the higher the refractive index obtained, the better the quality of the essential oil obtained. Obtained essential oil with the best index of refraction that is in the sample with a random sample size and distillation time of 5 hours.

Composition Analysis of Fragrant Citronella Oil with GC-MS tool



Peak Report TIC						
Peak#	R.Time	Area	Area%	Height	Height%	A/H Name
1	3.432	40772483	46.77	3064429	24.20	13.31 6-Octenal, 3,7-dimethyl-, (R)-
2	3.919	9029261	10.36	1211753	9.57	7.451 beta-Citronellol
3	4.091	18803410	21.57	2238904	17.68	8.40 trans-Geraniol
4	4.778	1751286	2.01	336127	2.65	5.21 6-Octen-1-ol, 3,7-dimethyl-, acetate
5	5.242	1616776	1.85	363999	2.87	4.441 BETA, ELEMENTE
6	5.497	1039265	1.19	213935	1.69	4.861 Caryophyllene
7	6.138	1811570	2.08	582174	4.60	3.111 delta-Cadinene
8	6.289	8300166	9.52	3186705	25.17	2.60 Cyclohexanemethanol, 4-ethenyl-, alpha-, al
9	6.934	1173044	1.35	448426	3.54	2.62 1-Naphthalenol, 1,2,3,4,6a,7,8,8a-octa
10	7.015	2880290	3.30	1015849	8.02	2.841 alpha-Cadinol
		87177491	100.00	12662301	100.00	

Figure 1.4 Results of GC-MS Analysis of Fragrant Citronella Oil

From the results of the GC-MS analysis it can be seen that the chemical compounds identified were 21.57% Geraniol and 10.36% Citronellol. Geraniol levels in essential oils are usually used for making perfume.

4 CONCLUSION

In this study it can be concluded that the distillation of citronella oil by vacuum hydrodistillation method is very effective. Seen from the results of the analysis of essential oils obtained. For the best distillation time for 5 hours of distillation with random pieces.

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