

INFLUENCE OF SOLVENT TYPE ON PATCHOULI OIL EXTRACTION EFFICIENCY USING MICROWAVE-ASSISTED METHOD

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Abstract

Nilam (*Pogostemon cablin Benth.*) is an essential oil-producing plant with high economic value. Patchouli oil consists of components with a high boiling point, so it is very well used as a binding agent in the perfume industry. Patchouli oil is also used in the cosmetics and perfume industry, as well as in the food and beverage industry and the pharmaceutical industry. This study aims to determine the effect of solvent type and extraction time on the obtained patchouli oil yield and the composition of patchouli oil through gas chromatography-mass spectrometry (GC-MS) analysis. Patchouli leaves were extracted using the microwave-assisted extraction (MAE) method at 375 W microwave power with 30, 45, 60, 75, and 90-minute time variations. The solvents used in this study were hexane and ethanol with a raw material to solvent ratio of 1:15. The results showed that patchouli oil yield increased with increasing extraction time until it reached the optimum value. However, after reaching the optimum value, the yield decreased with increasing time. Patchouli oil with ethanol has the highest yield at 75 minutes of extraction time, with a yield of 28.45%, and patchouli oil with hexane has the highest yield at 45 minutes, which is 6.25%. Based on GC-MS test, patchouli oil contains patchouli alcohol, trans-caryophyllene, α -guaiene, α -patchoulene, β -patchoulene, δ -guaiene and veridiflorol. The patchouli alcohol (PA) content contained in patchouli oil with ethanol is 5.192%, while patchouli oil with hexane contains patchouli alcohol of 2.445%.

Keywords: Extraction; Microwave; Patchouli alcohol; Patchouli oil

1. INTRODUCTION

Indonesia has a high prospect for the export commodity of patchouli oil, which boasts a high export value and contributes approximately 60% to the total national essential oil exports (Hernawati & Budiarti, 2012). Approximately 90% of the world's patchouli oil is supplied by Indonesia (Arpi et al., 2011). This essential oil plays a significant role in the cosmetics and perfume industry, food and beverage industry, and pharmaceutical industry and serves as a binding agent (Kamar, 2019).

Indonesia is one of the world's largest producers of patchouli oil (Rifai et al., 2019). The main export destinations for patchouli oil are the USA, France, Germany, the UK, Belgium, Singapore, Switzerland, and India (Lestari et al., 2023). Most of the patchouli oil in Indonesia is produced in Aceh, North Sumatra, and

West Sumatra. Other regions that produce patchouli oil include Lampung Bengkulu and are rapidly developing in Java as well as in eastern Indonesia, such as Kalimantan and Sulawesi (Sahwalita & Herdiana, 2016).

The demand for patchouli oil is constantly increasing, presenting a significant opportunity for Indonesia (Idris et al., 2014). The global demand for patchouli oil is around 1,200 tons annually, with a 5% growth rate. Meanwhile, patchouli production in Indonesia is 2,382 tons, and most of the oil is exported for use in fragrances, cosmetics, antiseptics, and insecticides (Hariyani et al., 2015; Prawoto & Sholeh, 2006).

Extraction separates a mixture of several substances into its components (Amiarsih et al., 2016). A more efficient method has been discovered for

patchouli leaf extraction, namely the microwave-assisted extraction (MAE) method. The MAE method has advantages such as a very short extraction time and produces high product purity levels and yield (Kartika Fitri & Widyastuti, 2017). Based on previous research by Yulistiani et al. (2020), spearmint leaf extraction using MAE with a power of 100, 180, and 300 W and extraction times of 5, 10, 15, and 20 minutes were studied. Spearmint leaf oil yield increased with increasing power. The highest yield obtained was 5.17% when the power used was 180 watts with an extraction time of 15 minutes. Therefore, MAE has advantages in extracting leaves quickly while obtaining good quality (Yulistiani et al., 2020).

The quality of patchouli oil obtained from Nilam leaf extraction is also influenced by the pre-treatment of the material before extraction (drying method). The drying process aims to reduce the moisture content in patchouli leaves. Ardianto et al., (2019) showed that the patchouli alcohol content obtained was 30%, which meets the Indonesian National Standard (SNI) requirements. Therefore, factors such as drying method and drying time affect the quality of the patchouli oil itself, such as moisture content of patchouli leaves, aroma, color, specific gravity, refractive index, acid number, ester number, and solubility in alcohol of the patchouli oil, but does not have a significant effect on yield, color and optical rotation of the patchouli oil (Lubis et al., 2022).

Selecting the type of solvent, there are several things to consider, such as a solvent that can absorb microwave energy and has a high dielectric constant number because the amount of energy absorbed must be proportional to its dielectric constant (Liompart et al., 2018; Foong, 2008). Based on research by Adiyasa et al. (2015), the average yield value of essential oil extraction from mandarin orange peel using ethanol was 47.69% higher than that of n-hexane at 3.34%. This is due to the polarity of compounds contained in mandarin orange peel having polarity close to the polarity of the ethanol so that the number of compounds extracted by ethanol is greater than the n-hexane (Adiyasa et al., 2015).

This research investigates the effect of extraction time and solvent on patchouli oil production from patchouli leaf.

2. MATERIALS AND METHOD

2.1 Material

The materials used in this research are patchouli leaves from Bandung, West Java; two solvents are used, namely 99% technical grade n-hexane with the ProShield brand and 96% technical grade ethanol with the Eproduk brand.

2.2 Extraction of Patchouli Leaves

The Nilam leaves were cleaned from the stems and dried using a Memmert oven at 45°C for ±11 hours. Subsequently, the extraction was performed using a sharp microwave oven modified for MAE operation, as shown in Figure 1. Twenty-five grams of dried patchouli leaves, wrapped in filter paper, and 375 mL

of solvent (the ratio between the mass of raw material and the volume of solvent being 1:15 w/v) were placed into a 1000 mL three-neck flask. The solvents used for the extraction consisted of n-hexane and ethanol. The extraction was performed at a microwave power of 375 W with varying times of 30, 45, 60, 75, and 90 minutes. After the extraction stage, the oil-solvent mixture was filtered using filter paper. Then, a separation process was carried out using a rotary evaporator to remove a large amount of solvent from the patchouli oil. The separation was conducted at a temperature of 50°C with a rotation speed of 80 rpm and a vacuum pressure of 335 mbar for n-hexane and 175 mbar for ethanol.

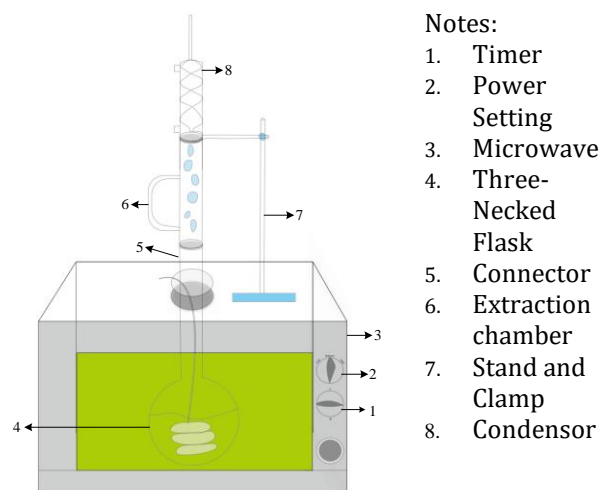


Figure 1. Microwave-assisted extraction (MAE) apparatus

Notes:

1. Timer
2. Power Setting
3. Microwave
4. Three-Necked Flask
5. Connector
6. Extraction chamber
7. Stand and Clamp
8. Condensor

2.2.1 Preparation of raw material

Patchouli leaves were dried as raw material in a Memmert oven at 45°C for ±11 hours. The dried leaves were ground using a blender and sieved with a 30-mesh sieve. The moisture content was determined using Equation 1.

$$M = \left(\frac{W_o - W_d}{W_o} \right) \times 100\% \quad (1)$$

Where M is the moisture content (%), W_o is the mass of fresh patchouli leaves (g), and W_d is the mass of dried patchouli leaves (g).

2.2.2 Yield analysis

The patchouli oil that was separated from the solvent and placed in a glass bottle was then weighed, and the yield percentage produced was calculated using Equation 2.

$$\text{yield (\%)} = \frac{\text{Mass of Patchouli Oil Extract}}{\text{Mass of Sample}} \times 100\% \quad (2)$$

2.2.3 Specific gravity analysis

Specific gravity is the ratio of the oil's weight to the water's at the same volume and temperature. The instrument used for measuring the oil's specific gravity is a pycnometer. First, the pycnometer was cleaned with alcohol, then dried and weighed. After that, it was filled with distilled water or oil and weighed again. The specific gravity can be calculated using equation 3.

$$\text{Specific gravity} = d_{25}^{25} = \frac{m_2 - m}{m_1 - m} \quad (3)$$

where m is the mass of the empty pycnometer (g); m_1 is the mass of the pycnometer filled with water at 25°C (g); and m_2 is the mass of the pycnometer filled with oil at 25°C (g).

2.2.4 Gas chromatography-mass spectrometry (GC-MS) analysis

In this phase, the evaporated patchouli oil with the highest yield value and specific gravity close to the Indonesian National Standard (SNI) was analyzed using Gas Chromatography-Mass Spectrometry (GC-MS). The GC-MS analysis was conducted to measure the types and content of compounds in the extracted patchouli oil, both quantitatively and qualitatively. The study was performed using GC-MS with a retention time of 30 minutes.

3. RESULTS AND DISCUSSION

3.1 Extraction Results of Patchouli Leaves

In this study, as shown in Table 1, microwave-assisted extraction (MAE) of patchouli oil using a combination of ethanol and hexane as solvents yielded 28.45% and 6.25%, respectively. Compared to previous studies, this MAE method achieved significantly higher efficiency than traditional techniques, such as steam

distillation, which only produced a 0.71% yield (Soh et al., 2020), and microwave hydrodistillation (MAHD), which yielded 2.33% (Abdurahman & Sundarajan, 2019). While supercritical fluid extraction (SFE) yielded a comparable 3.51% (Soh et al., 2020). The simplicity, shorter extraction time, and high yield of the MAE method in this study suggest it is a more effective alternative for extracting patchouli oil. This demonstrates that combining MAE with specific solvent choices can significantly enhance oil yield, making it a promising approach for future applications in essential oil extraction.

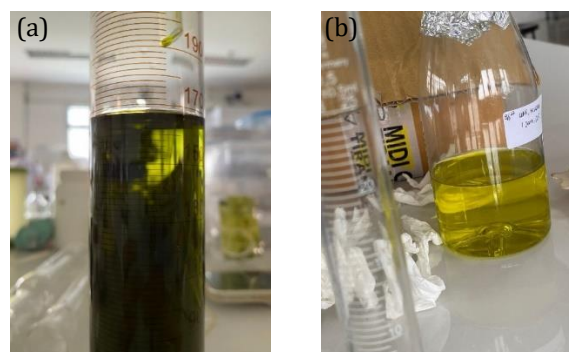


Figure 2. Comparison of patchouli leaf extraction results with (a) ethanol and (b) hexane

Table 1. Comparison of patchouli oil with various extraction methods

Materials	Technology	Operating Conditions	Yield	Ref.
<ul style="list-style-type: none"> • Patchouli Leaves • Acetone, Ethanol, Hexane 	Maceration	Ratio: 1:10 w/v, soaking time: overnight	<ul style="list-style-type: none"> • Acetone: 8,34% • Ethanol: 30,99% • Hexane: 13,15% 	(Adam et al., 2014)
<ul style="list-style-type: none"> • Patchouli Leaves • Hexane 	Soxhlet	Ratio: 1:8.3 w/v, t: 8 hours	7,4741%	(Syahputra et al., 2017)
<ul style="list-style-type: none"> • Patchouli Leaves • Water 	Microwave Assisted Hydrodistillation (MAHD)	Ratio: 1:12 w/v, t: 45 minutes, power: 400 W	2,33%	(Abdurahman & Sundarajan, 2019)
<ul style="list-style-type: none"> • Patchouli Leaves • Water 	Hydrodistillation (HD)	Ratio: 1:12 w/v, t: 45 minutes	0,9%	(Abdurahman & Sundarajan, 2019)
<ul style="list-style-type: none"> • Leaves and stems of patchouli • Liquid CO₂ 	Supercritical Fluid Extraction (SFE)	Ratio: 1:12 w/v, t: 45 minutes, T: 45°C, P: 15 MPa, t: 150 minutes, CO ₂ flow rate: 60 g/minute	3,51%	(Soh et al., 2020)
<ul style="list-style-type: none"> • Leaves and stems of patchouli • Water 	Steam distillation	T: 100°C, t: 5 hours	0,71%	(Soh et al., 2020)
<ul style="list-style-type: none"> • Nilam Leaves • Ethanol, hexane 	Microwave Assisted Extraction (MAE)	Ratio: 1:15 w/v, power: 375 W, t_{etanol} : 75 minutes, t_{heksana} : 45 minutes	<ul style="list-style-type: none"> • Ethanol: 28,45% • Hexane: 6,25% 	This study

Figure 2 shows the extraction results of patchouli leaves from hexane and ethanol, which produce different colors. The hexane produces a yellowish-green color, while the ethanol produces a dark green color. One of the factors why the ethanol is dark green is due to the presence of coloring substances in the form of tannins and oil compounds from the leaves that are soluble in organic solvents (Hidayati & Khaerunisa, 2018). With the use of hexane, which has a yellowish-green color, means only a small amount of chlorophyll can be extracted (Firyanto et al., 2020).

3.2 The Influence of Solvent on Patchouli Oil Yield

Regarding the extraction of patchouli leaves, the efficiency of the solvents is assessed for their ability to extract components from the original material (Adam et al., 2019). Figure 3 presents data derived from two variations of solvent extraction experiments conducted twice.

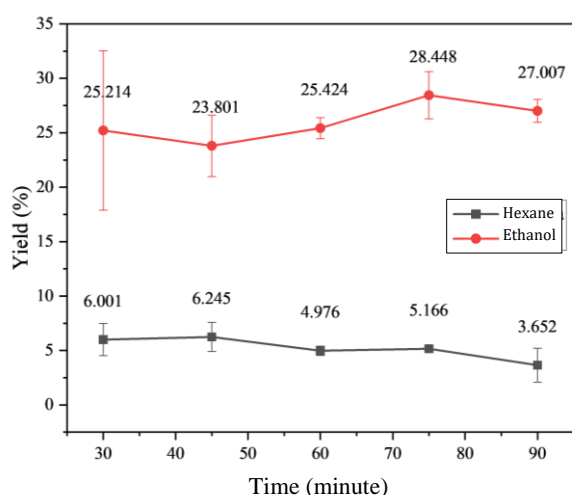


Figure 3. The influence of time on patchouli oil yield

Based on the results of this study, the highest yield was obtained from ethanol, and the lowest yield was obtained from hexane. The highest average yield from ethanol was 28.45%, while the highest yield from hexane was 6.25%. Then, the lowest average yields obtained from ethanol and hexane were 23.80% and 3.65%, respectively. According to Muhamad's 2019 research, these results are influenced by the polarity of each solvent and its ability to attract all types of active

compounds, such as polar and non-polar (Muhamad et al., 2019). All solvents can penetrate cell walls depending on the compatibility of the material to be extracted, in accordance with the principle of like dissolves like (Yulianti et al., 2021). Ethanol, a polar compound, shows the highest percent yield, meaning the polarity of the compounds in the patchouli leaf extract is close to the polarity of the ethanol compared to hexane, which is a non-polar compound (Yulianti et al., 2021; Kusumaningrum et al., 2023). Ethanol has a higher dielectric constant value than hexane. Ethanol has a dielectric constant value of 24,3, while the dielectric constant value of hexane is 1.89 (Liompert et al., 2018).

3.3 The Influence of Time on Patchouli Oil Yield

The results will generally increase with increasing extraction time until reaching an optimum value (Veggi et al., 2013). Based on Table 1 and Figure 3, it can be seen that patchouli oil with ethanol has the highest yield at 75 minutes (28.45%), and patchouli oil with hexane has the highest yield at 45 minutes (6.25%). The patchouli oil yield decreased with increasing time. Similar results occurred in research conducted by Abdurahman and Sundarajan (2019), which showed that the results of patchouli leaf extraction using microwave-assisted hydrodistillation (MAHD) reached the highest yield after extraction was carried out for 45 minutes with a yield of 2.33% and then the yield decreased to 1% with increasing time (Abdurahman & Sundarajan, 2019).

Each solvent has a different optimal extraction time. For hexane, the optimal time is 45 minutes of extraction, while ethanol reaches its optimal time at 75 minutes. However, ethanol still produces a higher yield than hexane, as shown in Figure 3. Based on the GC-MS results, the amount of patchouli alcohol extracted using ethanol was 47% higher than that extracted using hexane. Patchouli alcohol contains a hydroxyl group (OH-), which easily dissolves in ethanol due to its polar nature, resulting in a greater extraction yield (Kok et al., 2023).

3.4 Chemical Composition of Patchouli Oil

Some crude patchouli oil component analysis results can be seen in Table 2, Figure 4, and Figure 5. In research conducted by Souhoka et al. (2020), it was

Table 2. The Composition of Patchouli Oil Compounds

Component	Chemical formula	Result			
		N-Hexane		Ethanol	
		RT (minute)	Percentage (%)	RT (minute)	Percentage (%)
<i>Trans-Caryophyllene</i>	C ₁₅ H ₂₄	17,914	0,448	-	-
<i>β-patchoulene</i>	C ₁₅ H ₂₄	-	-	17,564	0,082
<i>α-guaiene</i>	C ₁₅ H ₂₄	18,014	0,116	17,974	1,137
<i>α-patchoulene</i>	C ₁₅ H ₂₄	18,299	0,192	18,259	0,343
<i>δ-guaiene</i>	C ₁₅ H ₂₄	19,819	0,188	18,524	1,686
<i>Veridiflorol</i>	C ₁₅ H ₂₆	-	-	19,679	0,320
<i>Patchouli alcohol</i>	C ₁₅ H ₂₆ O	19,975	2,445	19,834	5,192
<i>Azulene</i>	C ₁₀ H ₈	-	-	-	-
<i>Seychellene</i>	C ₁₅ H ₂₄	-	-	-	-

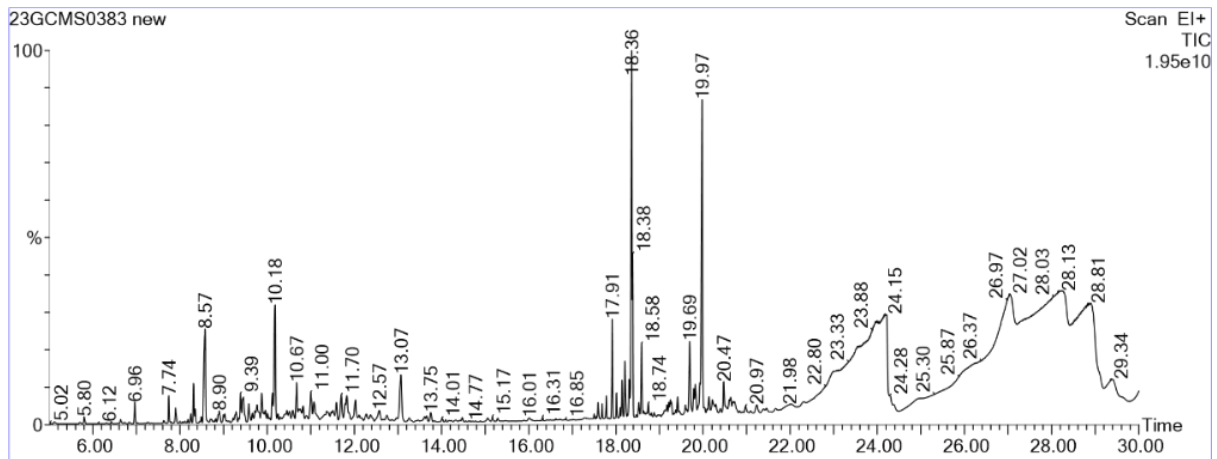


Figure 4. Chromatogram of patchouli oil with hexane solvent

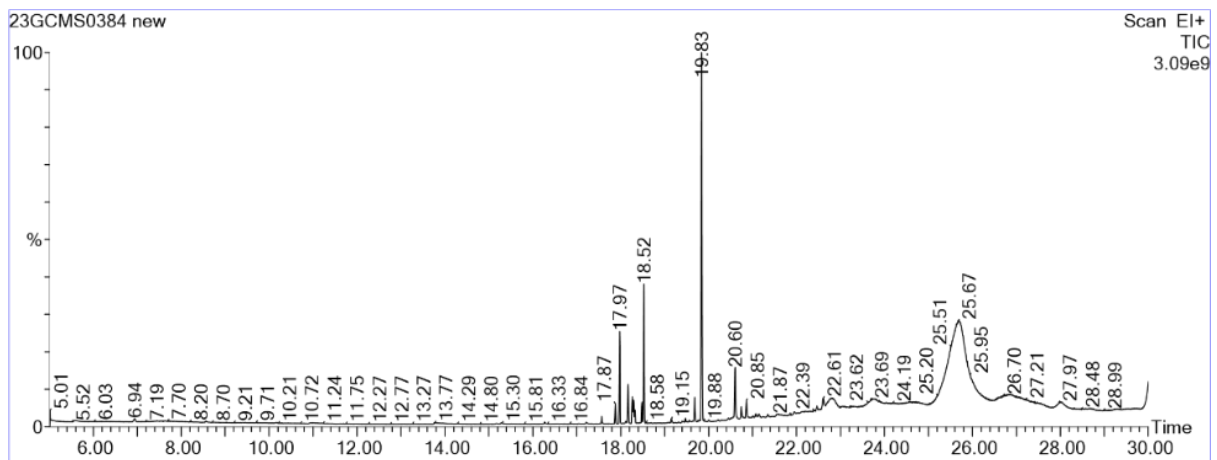


Figure 5. Chromatogram of patchouli oil with ethanol solvent

shown that patchouli alcohol (22.70%), δ -guanine (18.90%), α -guanine (18.61), azulene (8.74), trans-caryophyllene (7.96%), Seychelles (5.70%), β -patchouli (4.56%) and veridiflorol (4.39) are patchouli oil components that have a large percentage (Souhoka et al., 2020). Although the GC-MS analysis results indicate the presence of components, as mentioned in this study, these components have low percentages. To enhance the purity of patchouli oil, vacuum fractionation distillation can be used, as it allows for the separation of components at lower temperatures, reducing thermal degradation and preserving the quality of the essential oil (Rifai et al., 2019).

According to Syarifatuz Zaimah (2014), there are factors that cause the PA levels to be low, and it is likely that damage has occurred to the chemical compounds of patchouli oil. This damage can be caused by hydrolysis, oxidation, resinification, mixing of oil with other materials, and improper storage of patchouli oil,

which can also affect damage to chemical compounds in patchouli oil.

The composition of the chemical components that make up patchouli oil both qualitatively and quantitatively, apart from being influenced by the conditions of the extraction process, can also be influenced by factors such as plant origin, environmental conditions, cultivation conditions, harvest time and post-harvest processing (Souhoka et al., 2020; Kok et al., 2023).

3.5 Cost Analysis

Based on Table 3, the main constraints in the development of this extraction process are equipment costs, with the member's oven for 40.13% - 40.27% of the total price, the microwave at 16.38% - 16.44%, and labor costs with employee salaries at 29.48% - 29.59% (Peters & Timmerhaus, 2018).

Table 3. Cost Analysis of Microwave-Assisted Extraction

Cost	N-Hexane		Ethanol	
	Annual Cost (Rp)	Total percentage	Annual Cost (Rp)	Total percentage
1. Fixed Cost				
a. Material:				
Microwave set	10.000.000	16.38%	10.000.000	16.44%
Mesh	190.000	0.31%	190.000	0.31%
Thermometer	150.000	0.25%	150.000	0.25%
Hose 8 meter	17.000	0.03%	17.000	0.03%
Rotary Evaporator	5.133.333	8.41%	5.133.333	8.44%
Menmert Oven	24.500.000	40.13%	24.500.000	40.27%
Hose	32.000	0.05%	32.000	0.05%
Glass Funnel 100 mm	37.000	0.06%	37.000	0.06%
Measuring Glass 500 mL	115.000	0.19%	115.000	0.19%
Beaker Glass 500 mL	45.000	0.07%	45.000	0.07%
Pycnometer 1 mL	55.000	0.09%	55.000	0.09%
Water collection basin	80.000	0.13%	80.000	0.13%
Blender 2L	54.800	0.09%	54.800	0.09%
Total equipment cost	40.409.133		40.409.133	
b. Monthly labor salary	18.000.000	29.48%	18.000.000	29.59%
Total annual fixed cost	58.409.133		58.409.133	
2. Variable Cost				
a. Consumables				
Solvent	316.000	0.52%	98.000	0.16%
Patchouli leaves	896.000	1.47%	896.000	1.47%
Nylon rope	9.000	0.01%	9.000	0.01%
Filter paper	68.500	0.11%	68.500	0.11%
Cloth strainer	33.000	0.05%	33.000	0.05%
Bottles 100 mL & 350 mL	25.000	0.04%	25.000	0.04%
Aluminum foil	53.000	0.09%	53.000	0.09%
Ziplock bags	32.000	0.05%	32.000	0.05%
Label Paper	7.500	0.01%	7.500	0.01%
Glass bottles 10 mL	59.000	0.10%	59.000	0.10%
Plastic bottles 250 mL	35.500	0.06%	35.500	0.06%
b. Electricity				
Oven	300.000	0.49%	300.000	0.49%
Microwave	200.000	0.33%	200.000	0.33%
Rotary Evaporator	70.000	0.11%	70.000	0.12%
c. Water				
Tap Water	540.000		540.000	0.89%
Total Variable Cost	2.644.500		2.426.500	
Total cost	61.053.633		60.835.633	
<i>Production cost per kg</i>	508.780		506.964	

4. CONCLUSION

Regarding the influence of solvent on yield, the highest average yield for ethanol was 28.45%, and for hexane was 6.25%. This is because a compound will be based on the similarity of polarity with its solvent. Therefore, polar compounds will dissolve in polar solvents. The yield of patchouli oil increased with increasing time. It decreased after the patchouli oil reached the highest yield, with ethanol obtained at an extraction time of 75 minutes and hexane obtained at an extraction time of 45 minutes. The extract contained components such as patchouli alcohol (5.192% for ethanol and 2.445% for hexane), trans-caryophyllene, α -guaiene, α , β -patchoulene, δ -guaiene/ α -bulnesene, and veridiflorol, despite the low concentrations of these components.

5. ACKNOWLEDGMENT

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