A Preliminary Study of Patchouli Oil Extraction by Microwave Air-Hydrodistillation Method

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Abstract – Patchouli oil extraction in general is still using conventional methods that require a long time of extraction. It is therefore necessary to develop extraction methods to obtain patchouli oil with optimum yield and quality. One of the new methods, which has been successfully developed, is microwave hydrodistillation (MHD). In addition to optimizing the extraction process of patchouli oil, this study also used microwave air-hydrodistillation (MAHD). Based on the research results, extraction using MAHD method can produce higher yield of patchouli oil when compared using MHD method. Also, based on the results of the analysis by GC-MS, extraction using MAHD method can produce quality of patchouli oil that is almost the same when compared using MHD method. This is supported by the results of the analysis by GC-MS, which showed that the content of patchouli alcohol is the main component of patchouli oil, and is almost the same for patchouli oil extracted using MHD method (26.32%) and MAHD method (25.23%).

Key words: Air flow, Microwave hydrodistillation, Microwave air-hydrodistillation, Patchouli oil, Pogostemon cablin Benth

1. Introduction

Patchouli is one of the essential oil producing plants in Indonesia and has a high economic value. Indonesia is largest patchouli oil supplier in the world market, with nearly 70% of total world patchouli oil demand. Another producer of patchouli is China [1]. In addition, the type of patchouli for the variety of *Pogostemon cablin* Benth, originally from the Philippines, later expanded into Malaysia and Indonesia.

The extraction of essential oil in Indonesia has existed since colonial times. But the quality and quantity of produced essential oils in general is still not good, because most of the essential oils extraction still uses conventional methods. The extraction of patchouli oil in Indonesia is generally still using steam distillation method. Extraction with conventional methods only produces patchouli oil of low yield and quality. Additionally, at this time extraction of patchouli oil using conventional methods requires a long time of extraction. It is therefore necessary for the development of the extraction methods to obtain patchouli oil with optimum yield and quality.

One of the new methods that has now been successfully developed is extraction with microwave-assisted extraction (MAE). This is supported by some previous studies that demonstrate MAE method is one of the new methods that has potential to be developed. Currently, one of the MAE methods that have been successfully developed is microwave hydrodistillation (MHD) [2]. In this study, extraction of patchouli oil was done using MHD method. In addition to optimizing the extraction process of patchouli oil, this study also used the development of MHD method called microwave air-hydrodistillation (MAHD) method. Previous research by Kusuma and Mahfud (2017b) [3] showed that the extraction of patchouli oil with MAHD method can produce yield and recovery higher when compared with MHD method. In addition, research by Kusuma and Mahfud (2017b) [3] also showed that the extraction of patchouli oil using MAHD method can produce patchouli oil that has heavy fraction with higher number when compared using MHD method.

Therefore, we studied some parameters that affect the extraction of patchouli oil by using MHD and MAHD methods. Until now, the various previous research has not studied the parameters that affect the extraction of patchouli oil using MAHD method.

2. Material and Methods

2-1. Materials and chemicals

Patchouli (*Pogostemon cablin* Benth) leaves used in this study were obtained from Trenggalek, East Java, Indonesia. Patchouli leaves are used as dried leaves in the form of intact (6.50 ± 0.62 cm). Aquadest and anhydrous sodium sulfate used in the experimental work were all of analytical grade.

2-2. Microwave air-hydrodistillation (MAHD)

Domestic microwave (EMM-2007X, Electrolux, 20 L, max microwave power of 800 W, 2.45 GHz), which has dimensions of 46.1 cm \times 28.0 cm \times 37.3 cm was modified prior to operating MAHD. First, the patchouli (*Pogostemon cablin* Benth) leaves were weighed according to the ratio of feed to solvent (F/S) (0.05, 0.10 and 0.15 g/mL). Distilled water was used as the solvent with volume of 400 mL. Patchouli leaves and distilled water were put into a distiller in the

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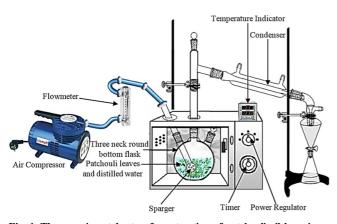


Fig. 1. The experimental setup for extraction of patchouli oil by microwave air-hydrodistillation method.

form of three-neck round bottom flask. The addition of air flow on MAHD method was performed using a compressor (Melzer V-777, electric motors: 1/5 H.P., max pressure: 3 bar). The air flow rate used was 1.5 L/min and 3.0 L/min. While the microwave power was 264 W, 400 W and 600 W. The extraction of patchouli oil used MAHD method for two hours. The obtained patchouli oil was then separated from distilled water with a separatory funnel. The remaining distilled water was then removed by adding anhydrous sodium sulfate on patchouli oil that had been obtained. Patchouli oil was then weighed and stored in vials at temperature around 4 °C. Yield of obtained patchouli oil was calculated using the following equation:

Yield (%) = $\frac{\text{mass of patchouli oil (g)}}{\text{mass of patchuli leaves (g)}} \times 100$

2-3. Microwave hydrodistillation (MHD)

Domestic microwave (EMM-2007X, Electrolux, 20 L, max microwave power of 800 W, 2.45 GHz), which has dimensions of 46.1 cm \times 28.0 cm \times 37.3 cm, was modified prior to operating MHD. First, the patchouli (*Pogostemon cablin* Benth) leaves were weighed according to the ratio of feed to solvent (F/S) used (0.05, 0.10 and 0.15 g/mL). Distilled water was used as the solvent with volume of 400 mL.

Two neck round bottom flask Patchouli leaves and distilled water

Fig. 2. The experimental setup for extraction of patchouli oil by microwave hydrodistillation method.

Patchouli leaves and distilled water were put into a distiller in the form of two-neck round bottom flask. The microwave power used was 264 W, 400 W and 600 W. The extraction of patchouli oil used the MHD method for 2 hours. The obtained patchouli oil was then separated from distilled water by separatory funnel. The remaining distilled water was then removed by adding anhydrous sodium sulfate on patchouli oil that had been obtained. Patchouli oil was then weighed and stored in vial at temperature around 4 $^{\circ}$ C.

2-4. Chemical analysis of patchouli oil compounds

Patchouli oil composition was determined by gas chromatography coupled to mass spectrometry (GC-MS) analysis on a Hewlett-Packard 6890 gas chromatograph coupled to a 5973A mass spectrometer, using two fused-silica-capillary columns with different stationary phases. The non-polar column was HP5MS[™] (30 m length, 0.25 diameter and 0.25 µm film thickness) and the polar one was a Stabilwax[™] consisting of Carbowax[™]-PEG (60 m length, 0.25 mm diameter and 0.25 µm film thickness). GC-MS spectra were obtained using the following conditions: carrier gas He; flow rate 1.0 ml min⁻¹; split 1:50; injection volume 1.0 µL; injection temperature 300 °C; oven temperature was programmed from 100 to 250 °C at 10 °C min⁻¹; the mass spectra were recorded at an ionization voltage of 70 eV in electron ionization mode. Identification was confirmed by comparison of their mass spectral fragmentation patterns with those stored in the MS database (National Institute of Standards and Technology and Wiley libraries) and with mass spectra literature data [4,5]. For each compound on the chromatogram, the percentage of peak area relative to the total peak areas from all compounds was determined and reported as relative amount of that compound.

3. Results and Discussion

3-1. The effect of microwave power

In the patchouli oil extraction using MHD and MAHD methods can be seen that the microwave power has significant effect on the yield. The effect of microwave power on the yield of patchouli oil can be seen in Figures 3 and 4. The increase in yield of patchouli oil in accordance with the increase of the microwave power that is used

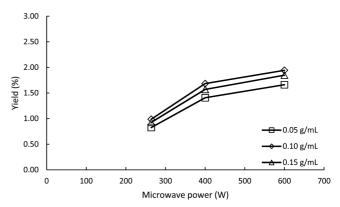


Fig. 3. The effect of microwave power on the extraction of patchouli oil using microwave hydrodistillation method.

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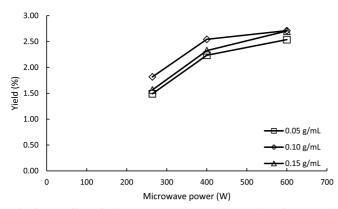


Fig. 4. The effect of microwave power on the extraction of patchouli oil using microwave air-hydrodistillation method (air flow rate 3.0 L/min).

due to the microwave power can affect the temperature of the system (material and solvent). This is because the higher supplied microwave power may cause the temperature of the system to rise rapidly. It is then causes the patchouli oil to be extracted more optimally with higher microwave power. This is consistent with previous research by Kusuma and Mahfud (2017a) [2], who extracted patchouli oil using MHD method with microwave power of 400 W and 600 W. The results of previous studies indicate that extraction of patchouli oil with microwave power of 600 W can produce higher yield of patchouli oil when compared with microwave power of 400 W.

3-2. The effect of feed-to-solvent ratio (F/S)

The feed-to-solvent ratio (F/S) is important because it will determine the number of samples that can be immersed in solvent. If the sample is not fully immersed in the solvent, the sample cannot be optimally extracted. Therefore, we also studied the effect of feed-tosolvent ratio (F/S) on the yield of patchouli oil extracted using MHD and MAHD methods. In general, the optimum yield of patchouli oil extracted using MHD and MAHD methods was obtained on feed-tosolvent ratio (F/S) of 0.10 g/mL. The effect of feed-to-solvent ratio (F/S) to the yield of patchouli oil can be seen in Figures 5 and 6.

3-3. The effect of air flow rate

In addition to the microwave power and feed-to-solvent ratio (F/

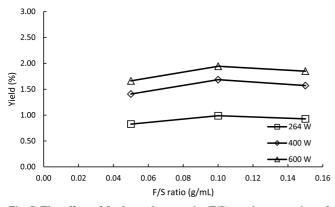


Fig. 5. The effect of feed-to-solvent ratio (F/S) on the extraction of patchouli oil using microwave hydrodistillation method.

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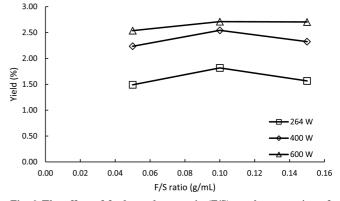


Fig. 6. The effect of feed-to-solvent ratio (F/S) on the extraction of patchouli oil using microwave air-hydrodistillation method (air flow rate 3.0 L/min).

S), the addition of air flow on the extraction of patchouli oil can affect the yield of obtained patchouli oil. In this study, a higher air flow rate was used, the yield of obtained patchouli oil became higher. The effect of used air flow rate to the yield of patchouli oil can be seen in Figures 7 and 8. The higher yield of obtained patchouli oil is because the addition of air flow can carry heavy patchouli oil component. Without the addition of air flow, the heavy component of patchouli oil cannot be optimally extracted. In addition, with more components of heavy oil that can be extracted also causes the yield of obtained patchouli oil to increase. This is in accordance with previous research

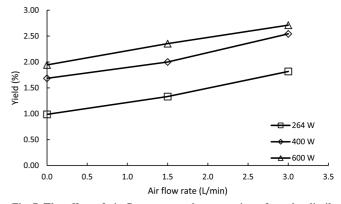


Fig. 7. The effect of air flow rate on the extraction of patchouli oil (F/S ratio of 0.10 g/mL).

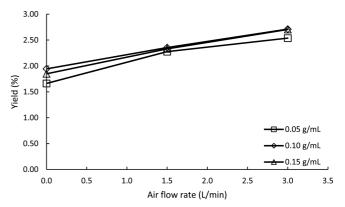


Fig. 8. The effect of air flow rate on the extraction of patchouli oil (microwave power of 600 W).

by Kusuma and Mahfud (2017b) [3], who extracted patchouli oil using MHD and MAHD methods. The results of previous studies indicate that extraction of patchouli oil using MAHD method can also higher yield of patchouli oil when compared with MHD method. Analysis using GC-MS can confirm the heavy component of patchouli oil that has been extracted by the addition of the air flow.

3-4. GC-MS analysis

GC-MS can confirm the heavy component of patchouli oil that has been extracted by the addition of air flow. From the analysis using GC-MS can be seen that the number of components of patchouli oil extracted using MAHD method (26 components) is more than when using MHD method (21 components). Therefore, it can be assumed that the addition of air flow on the extraction of patchouli oil can carry heavy component of patchouli oil. Furthermore, based on the analysis using GC-MS, the amount of oxygenated components of patchouli oil extracted using MAHD method (37.36%) is less when compared using MHD method (40.09%). Meanwhile, analysis using

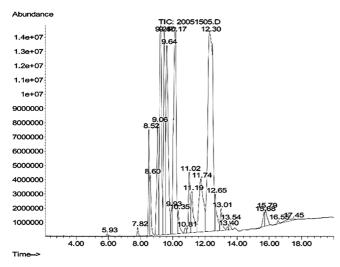


Fig. 9. The chromatogram of patchouli oil extracted using microwave hydrodistillation method.

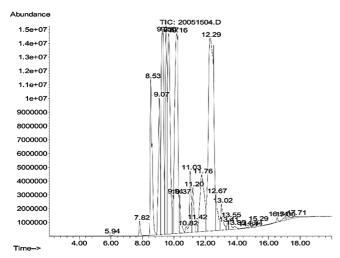


Fig. 10. The chromatogram of patchouli oil extracted using microwave air-hydrodistillation method.

GC-MS also showed that the content of patchouli alcohol is the main component of patchouli oil and is almost the same for patchouli oil extracted using MHD (26.32%) and MAHD (25.23%). So it can be said that the extraction of patchouli oil using MAHD method does not affect the quality of the obtained patchouli oil. A chromatogram of patchouli oil extracted using MHD and MAHD methods can be seen in Figures 9 and 10.

4. Conclusion

The extraction of patchouli oil using MHD and MAHD methods can be affected by parameters such as microwave power, feed-to-solvent ratio (F/S) and air flow rate. The optimum yield on extraction of patchouli oil using MHD and MAHD methods can be obtained at these operating conditions: microwave power of 600 W, feed-to-solvent ratio (F/S) of 0.10 g/mL and air flow rate of 3.0 L/min. The addition of air flow on the extraction of patchouli oil can carry heavy components of patchouli oil that are difficult to extract. This is supported by the results of analysis using GC-MS, which showed that the number of components of patchouli oil extracted using MAHD method (26 components) is more when compared using MHD method (21 components). Furthermore, analysis using GC-MS also showed that the content of patchouli alcohol is the main component of patchouli oil and is almost the same for patchouli oil extracted using MHD method (26.32%) and MAHD method (25.23%). So it can be said that the extraction of patchouli oil using MAHD method does not affect the quality of obtained patchouli oil.

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