Chiang Mai J. Sci. 2019; 46(4) : 741-755
http://epg.science.cmu.ac.th/ejournal/
Contributed Paper

Comparison of Microwave Hydrodistillation and Solvent-Free Microwave Extraction for Extraction of Agarwood Oil

Heri Septya Kusuma*, Ditta Kharisma Yolanda Putri, Isabel Triesty and Mahfud Mahfud
Department of Chemical Engineering, Faculty of Industrial Technology, Institut Teknologi Sepuluh Nopember, Surabaya 60111, Indonesia.
*Author for correspondence; e-mail: heriseptyakusuma@gmail.com; mahfud@chem-eng.its.ac.id

Received: 24 September 2018
Revised: 29 January 2019
Accepted: 11 February 2019

ABSTRACT

Production of agarwood (*Aquilaria malaccensis*) oil still potential to be developed in Indonesia. Agarwood oil was one of commodity that provides an important role for the country's foreign exchange earnings because the selling price was very high. However extraction of agarwood oil is currently still using the conventional method such as hydrodistillation which takes a long time to produce oil with good quality. In this research, the extraction of agarwood oil was done using microwave hydrodistillation and solvent-free microwave extraction method. The optimum conditions of the parameters that affected the agarwood oil extraction using both of methods as follows: microwave power of 450 W, ratio between mass of raw material with volume of distiller (F/D) of 0.01 g/mL, ratio between mass of raw material with volume of solvent (F/S) of 0.4 g/mL and raw materials that have powder size. Solvent-free microwave extraction method was an effective method to extract agarwood oil which with shorter extraction time can produced a higher yield of 5.95 times compared to microwave hydrodistillation method. In addition, based on the analysis of electric consumption and environmental impact, solvent-free microwave extraction method showed a smaller amount when compared with microwave hydrodistillation method. And also based on GC-MS analysis, the solvent-free microwave extraction method produces better quality of agarwood oil than the microwave hydrodistillation method. So based on that, the use of solvent-free microwave extraction method for agarwood oil extraction has been suitably used as a new green technique.

Keywords: agarwood oil, microwave hydrodistillation, solvent-free microwave extraction, green technique

1. INTRODUCTION

Indonesia has numerous and varied natural resources which are still cannot be used optimally. Among the biodiversity, plants that produce an essential oil are one of the natural resources which had not fully utilized. Indonesia produces 40-50 kinds of plant essential oil traded in the world and only some of the types of essential oils are entering the world market, including patchouli, lemongrass, cloves, jasmine, ylang, eucalyptus, sandalwood and vetiver.

Agarwood oil is a heavy oil that is still interesting to be developed. This is supported by the high selling price of agarwood oil that reaches 1,500 USD per tola (11.7 grams) of high quality oil [1]with its low wood price for 19 USD per kg and for high quality until it reaches 100,000 USD per kg [2]. Agarwood oil is also one of the commodities that provide an important role for the country's foreign exchange earnings from the total export of essential oils. However, the extraction of agarwood oil that has been done using conventional methods has less than maximum results. Where based on previous research, using 200 grams of agarwood powder and 2000 mL of water obtained oil yield is 0.18% using hydrodistillation method for 72 hours [3].

The length of time and the small yields obtained from these conventional methods, it is necessary to consider using a new "green technique" in the extraction of essential oils with minimum energy, solvent, and time usage. Several new methods have been developed to extract essential oils till now, one of which is by using microwave (microwave-assisted extraction). Previous research has shown that microwave extraction is an alternative that can be developed more than conventional methods, due to high levels of product purity, minimum solvent usage, and short processing times [4]. One of extraction method using microwave that has been successfully developed is microwave hydrodistillation method which is a combination of hydrodistillation with microwave heating [5]. Furthermore, microwave hydrodistillation method was developed again with solvent-free microwave extraction method. The method of solvent-free microwave extraction is an essential oil extraction method by using microwave heating, but does not require the addition of solvents such as microwave hydrodistillation method and other extraction methods [6]. It is expected to obtain higher agarwood oil yield with shorter extraction time if extraction is done using microwave hydrodistillation and solvent-free microwave extraction methods.

So based on that, agarwood oil will be carried out by using microwave hydrodistillation and solvent-free microwave extraction methods and then find out the effects of some parameters on extraction of agarwood oil using both of methods such as microwave power, raw material size, feed to solvent and feed to distiller ratio. It will also compare the yield of agarwood oil obtained, the necessary costs and the environmental impact of the agarwood oil extraction process using microwave hydrodistillation and solvent-free microwave extraction methods.

2. Materials and Methods

2.1 Materials and Chemicals

Agarwood (*Aquilaria malaccensis*) obtained from Merauke, Indonesia in the form of powder and chopped woods (± 2cm). Distilled water and n-hexane used in the experimental work were all of analytical grade.

2.2 Microwave Hydrodistillation Procedure

In employing microwave hydrodistillation, we used a domestic microwave oven (EMM-2308X, Electrolux, maximum delivered power of 800 W) with wave frequency of 2450 MHz. The dimensions of the PTFE-coated cavity of the microwave oven were 48.5 cm × 37.0 cm × 29.25 cm. The microwave oven was modified by drilling a hole at the top. A round bottom flask with a capacity of 1000 mL was placed inside the oven and was connected to clevenger condenser through the hole. Then, thehole was closed with PTFE to prevent any loss of the heat inside[7].

In the agarwood oil extraction using microwave hydrodistillation, distilled water (50 mL) and agarwood (powder and chopped size) were put in a series of round bottom flasks (1000 mL) to a ratio of feed to solvent

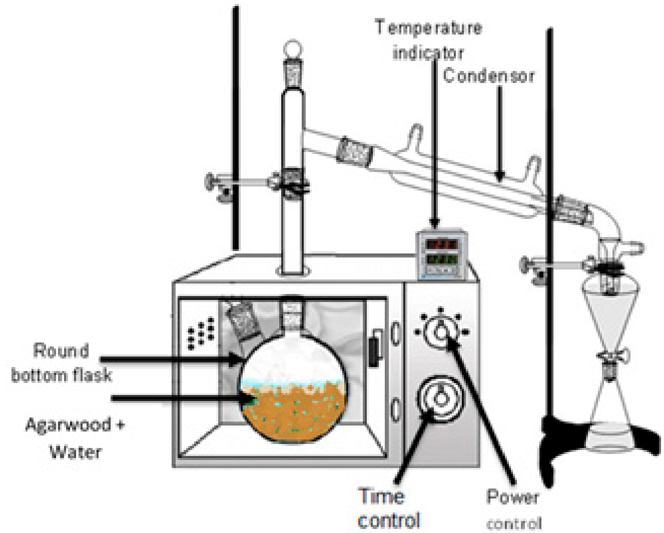


Figure 1. The experimental set up for extraction of essential oil from agarwood (*Aquilaria malaccensis*) using microwave hydrodistillation method.

of 0.2, 0.3, 0.4, and 0.5 g.mL^{-1}. The extraction was carried out for 12 hour under microwave power 300, 450, and 600 W. The extraction time shall be computed from the first drop out of the condenser. Essential oils were separated using a separating funnel that has been given n-hexane because the amount of agarwood oil was very little and then agarwood oil was obtained by evaporating n-hexane. After that, the obtained agarwood oil wasweighed and stored in amber vials at 4^{o}C until they were used for analysis.

2.3 Solvent-Free Microwave Extraction Procedure

Extraction of agarwood oil using solvent-free microwave extraction method was done more or less like microwave hydrodistillation method. But in this method, the materials were wetted before the extraction by soaking them in certain proportion of aquadest for 30 min and then removed the excess aquadest.In the agarwood oil extraction using solvent-free microwave extraction, agarwood (powder and chopped size) were put in a series of round bottom flasks (1000 mL) to a ratio of feed to distiller of 0.010, 0.015, 0.020, 0.025, and 0.030 g.mL^{-1}. The extraction was carried out for 6 hour under microwave power 300, 450, and 600 W.The agarwood oil was collected in amber vials and stored at 4^{o}C. The yield of agarwood oil was calculated as follows:

$$\text{Yield (\%)} = \frac{\text{Mass of extracted agarwood oil}}{\text{Mass of extracted agarwood}} \text{x}100\% \quad (1)$$

2.4 Electric Consumption

The electric consumption of the different extraction methods was calculated based on the infuence of power consumption and extraction time. The general equation for electric consumption is described by equation (2):

$$E_c = \frac{Pt}{1000} \quad (2)$$

where E_c is electric consumption (kW h), P is power consumption (W) and t is time (h). Additionally, the relative electric consumption of the different extraction methods could be expressed by equation (3):

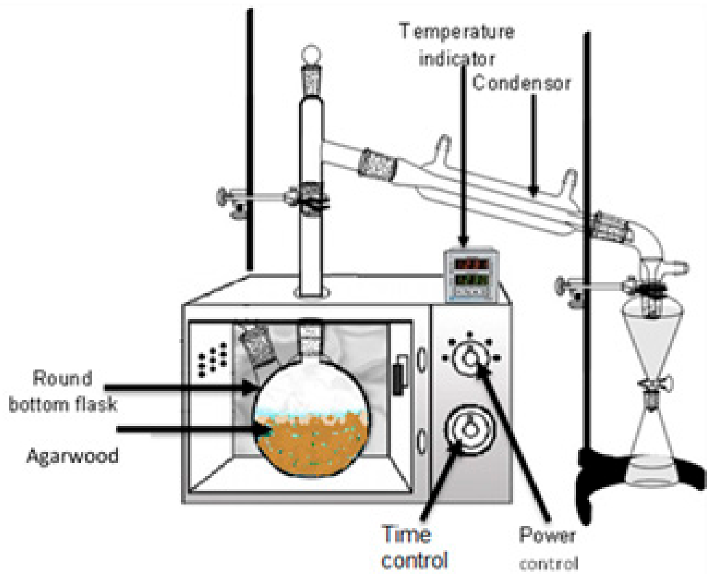


Figure 2. The experimental set up for extraction of essential oil from agarwood (*Aquilaria malaccensis*) using solvent-free microwave extraction method.

$$E_c^* = \frac{E_c}{m} \quad (3)$$

where E_c^* is relative electric consumption (kW h g^{-1}) and *m* is the mass of obtained essential oil (g) [8].

2.5 CO_2 Emission

The measurements of CO_2 emitted were carried out based on the procedures mentioned in previous studies: to obtain 1 kWh of energy from coal or fossil fuels, 800 g of CO_2 will be released into the atmosphere during combustion [4]. Thus CO_2 emission is described by equation (4):

$$E_{CO_2} = \frac{E_c 800}{1000} \quad (4)$$

where E_{CO_2} is CO_2 emission (g) and E_c is electric consumption (kWh).The relative CO_2 emission of the different extraction methods was calculated according to equation (5):

$$E_{CO_2}^* = \frac{E_{CO_2}}{m} \quad (5)$$

where $E_{CO_2}^*$ is the relative CO_2 emission (kg g^{-1}) and *m* is the mass of obtained essential oil (g) [8].

2.6 Chemical Analysis of Agarwood Oil Components by Gas Chromatography-Massspectrometry (GC-MS)

Components that contained in essential oils can be identified by analysis of GC-MS (Gas Chromatography-Mass Spectrometry). This method is an analytical method that combines gas chromatography and mass spectrophotometry methods so that components can be identified one by one in the sample. In addition to knowing the components contained in essential oils, this analysis can also be used to determine the levels for each component.

3. Results and Discussion

3.1 Effect of Microwave Power on Yield of Agarwood Oil

The microwave power used in the extraction process by using microwave hydrodistillation and solvent-free microwave extraction method is closely related to the process temperature, where the greater the power used, the system temperature at the extraction process will rapidly reach the boiling point of the solvent used. With the rapidly reaching boiling point of the solvent,it leads to increased yield of essential oils to reach insignificant conditions. In addition to extraction using microwave hydrodistillation and solvent-free microwave extraction methods, microwave power also acts as a driving force to break the cell membrane structure of the plant so that the oil can be diffused out and dissolved in the solvent. So the addition of microwave power in general will increase yield and speed up the extraction time [9].

Based on Figure 3, in this study generally can be seen that the most optimum microwave power is microwave power of 450 W that obtains agarwood oil yield of 1.3521%. Additionally in Figure 3 can be seen that the microwave power of 300 W produces a smaller yield when compared with the microwave power of 450 W. This is because the microwave power provided is still too small to cause damage to the oil glands so that the yield obtained becomes less than the maximum.Furthermore in microwave power of 600 W, there is a decrease in agarwood oil yield caused by the microwave power is given too large then the evaporation rate faster. This may lead to degradation of materials that can actually lower yields and could damage the essential oils that want to extract. As the study conducted by Chen et al. (2015),the microwave power consumption of 600 W causes the yield obtained to be smaller[10]. This is because if the power provided is too large then the evaporation rate will accelerate. This may lead to degradation of essential oil components.

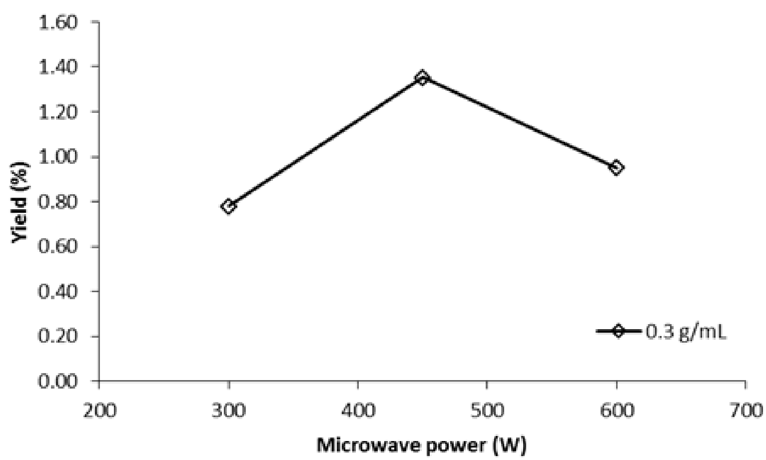


Figure 3. Effect of microwave power to yield extraction of agarwood oil using microwave hydrodistillation method (agarwood that has powder size, F/S ratio of 0.3 g/mL).

3.2 Effect of Ratio Between Mass of Raw Material with Volume of Distiller (F/D) on Yield of Agarwood Oil

In agarwood oil extraction using solvent-free microwave extraction method, the ratio between mass of raw material with volume of distiller (F/D) used in this study was 0.01; 0.015; 0.02, 0.025 and 0.03 g/mL. Where at that ratio, the mass of agarwood usedwas 10, 15, 20, 25 and 30 g. The effect of ratio between mass of raw material with volume of distiller (F/D) on yield of agarwood oil obtained using solvent-free microwave extraction method can be seen in Figure 4.

Based on Figure 4, it can be seen that the higher F/D ratio causes the obtained yield to be lower. The highest yield was obtained when the smallest F/D ratio (0.01 g/mL) of 1.6931%.

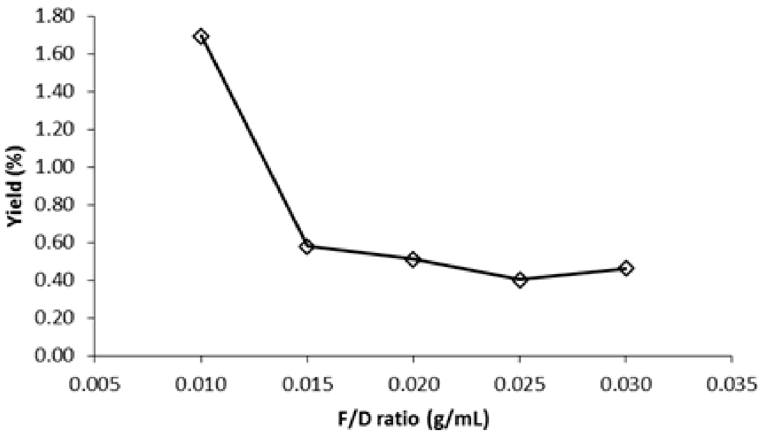


Figure 4. Effect of ratio between mass of raw material with volume of distiller (F/D) to yield for extraction of agarwood oil using solvent-free microwave extraction method (microwave power of 300 W and agarwood that has powder size).

This happens because at the smallest F/D ratio,agarwood can be extracted well with the density level of the material which is not too high. The density level of the material is closely related to the large space between materials. The density level of the material which is too high and even density of materials can lead to the formation of vapor paths "rat holes" that can decrease yield and quality of essential oils [11]. In addition,with the higher density level of the material will also lead to the rate of distillation or evaporation of essential oils that will be increasingly slow. This is due to the inhibition of steam movement to evaporateto the condenser, which can lead to reduced agarwood oil yield and decreases the efficiency of the distillation.

Besides due to the material density factor, this is also because the higher ratio used, the extraction load will be heavier so that the material is not extracted perfectly. Increasing the mass of materials used also causes the dielectric heating effect to be lower thereby decreasing the effects of microwave radiation that result in the extraction is not running optimally [12].

3.3 Effect of Ratio Between Mass of Raw Material with Volume of Solvent (F/S) on Yield of Agarwood Oil

One important factor affecting the extraction of essential oil using microwave hydrodistillation method is the selection of solvent. Selection of suitable solvents can make the extraction process more efficiently. In general, the capacity of the solvent to absorb microwave energy will be high if the solvent used has a high dielectric constant value. The dielectric constant value shows the ability of the solvent to be polarized by an external electric field and can be regarded as a relative measure of microwave energy density [13]. In addition, the dielectric constant also has functions to determine the interaction between the electric field and the matrix. So with the higher the dielectric constant value of a solvent, then the solvent will be better at absorbing the microwave energy. Therefore, in this study aquadest was used as a solvent. The selection of aquadest as a solvent in this study is also based on the previous study that aquadest has high dielectric constant [14].

The ratio between the raw materials to be extracted with the solvent is one of the important parameters that affect the yield of essential oil obtained. In general, the extraction of agarwood oil using microwave hydrodistillation method shows that the more raw materials used, the weight of agarwood oil obtained will increase. However, the large amount of raw materials and the amount of agarwood oil obtained, not always positively correlated with the increasing yield of agarwood oil obtained. This is because the yield of agarwood oil was influenced by the factor of ratio between agarwood oil obtained and the mass of initial raw material.

Based on Figure 5, it can be seen that the ratio obtained the highest yield on agarwood oil extraction using microwave hydrodistillation method with raw material that have powder size was 0.4 g/mL and decreased when using a higher F/S ratio of 0.5 g/mL. This is caused by the amount of solvent in the distiller in the extraction of agarwood oil using microwave hydrodistillation method. The ratio between the mass of material and the solvent used is very influential in agarwood oil yield where the amount of solvent in the distiller flask has the same amount in each process of agarwood oil extraction which is 50 ml, while the mass of the material used has increased every process of agarwood oil extraction. The ratio between the mass of material which has a rise with the same amount of solvent that causes decrease in yield.This phenomena was probably due to inadequate stirring of the solvent of the microwaves[15].

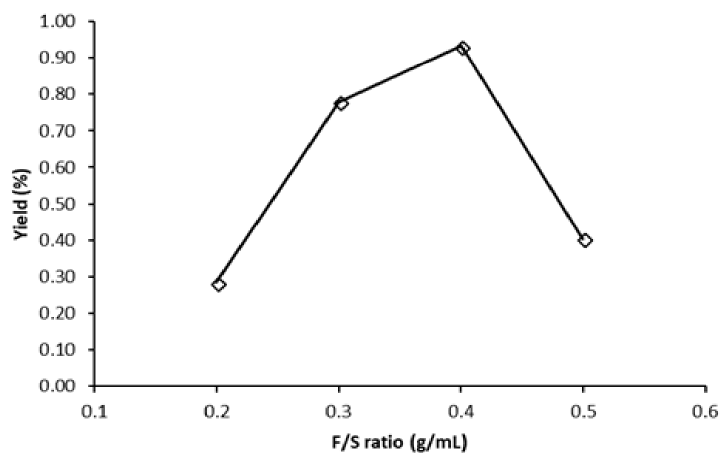


Figure 5. Effect of ratio between mass of raw material with volume of solvent (F/S) to yield for extraction of agarwood oil using microwave hydrodistillationmethod (microwave power of 300 W and agarwood that has powder size).

3.4 Effect of Raw Material Size on Yield of Agarwood Oil

In this study, the variation of agarwood size used was powder and chopped (± 2 cm). The effect of raw material size on yield of agarwood oil can be seen in Figure 6.

Based on Figure 6, it can be seen that the yield of agarwood oil obtained by using powder sized material is higher than using chopped sized material (± 2 cm) although the microwave power used is lower. This is due to the material size factor, where the powder material has a larger contact surface area than the chopping material so that the material will be extracted completely. The smaller size of this material is due to the cutting process being carried out due to the essential oil in the material surrounded by oil glands, vessels, oil

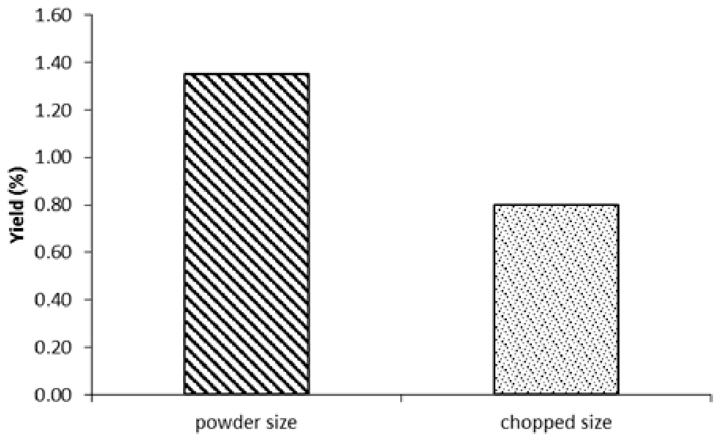


Figure 6. Effect of material size to yield extraction of agarwood oil using solvent-free microwave extraction (F/D ratio of 0.02 g/mL, microwave power of 300 W for powder size and 450 W for chopped size).

bags or glandular hair, so that when the material had intact size, the essential oil can only be extracted when water vapor succeeds through plant tissue and urged him to the surface. The cutting process also aims to keep the oil glands open as much as possible and the thickness of the plant material at the site of the diffusion will be reduced. Thus, when extracted, the evaporation rate of essential oils from plant material becomes faster [16].

The greater yield obtained from the powder material is also influenced by the water content factor. Where the amount of water content after soaking process for 30 min on the powder material is greater than the chopped material so that at the time of extraction process materials will not be easily burned and can be extracted optimally. Water content of powder and chopped materials after soaking process for 30 min can be seen in Table 1.

Table 1. Water content of agarwood after soaking for 30 min.

Material	Size	Water content (%)
Agarwood	Powder	86.248 ± 0.179
	Chopped (±2 cm)	83.508 ± 0.960

Based on previous research, it can also be seen that the amount of water content due to variations in immersion time also affects the yield of agarwood oil obtained. This can be seen in Table 2.

3.5 Comparison of Microwave Hydrodistillation and Solvent-Free Microwave Extraction Methodsin Agarwood Oil Extraction

This study has been carried out two extraction method, there were microwave hydrodistillation and solvent-free microwave extraction method. The difference between the two extraction methods was the extraction of agarwood oil using microwave hydrodistillationmethod used solvent in the form of distilled water, while for the extraction of agarwood oil using solvent-free microwave extraction method was not done the addition of solvent.

Based on Figure 7 it is known that with shorter extraction time, the yield of agarwood oil obtained by using solvent-free microwave

Table 2. The results of previous studies on agarwood oil extraction.

No.	Operating conditions of the process	Results	Reference
1.	Soaking time: 0, 7, 14, 21 and 28 days, conventional hydrodistillation method for 96 h, mass of *Aquilaria malaccensis*chips: 25 g; volume of solvent:125 L	The highest yield can be obtained by agarwood chips soaked for 14 days	[26]
2.	Agarwood tree of about 15 years old was selected and 15 kg matured woods were collectedfor the experiment. Woods were chopped into 2 to 3 inches long and 0.5 to 1 inch thin and then were soaked in water for 40 days. Extraction process using hydrodistillation method.	The yield of agarwood oil obtained was 0.0467 % (v/w)	[27]
3.	Agarwood samples were soaked in two solutions of 0.1 M lactic acid and 0.1 M sulphuric acid at room temperature (25 °C) for 168 hours. Then agarwood samples were dried for 24 hours in an oven at 50 °C.Extraction process using hydrodistillation method (mass of each sample was 200 gwith 1.5 L of water for 12 hours)	The yield of agarwood oil obtained were: unsoaked = 1.72%, soaked in lactic acid = 4.74%, and soaked in sulphuric acid = 6.78 %.	[28]
4.	Extraction of agarwood oil using hydrodistillation with extraction time 30 h. Several pretreatment methods performed on each sample prior to extraction were soaked with water pH 7, ethanol (50 % and 80 %), H_2SO_4 (pH 2 and 4), NaOH pH 10, mixed with enzyme for 120 h, and pretreatment with ultrasound for 30 h.	The highest yield obtained from pretreatment method with soaked in H_2SO_4 pH 2 of 0.22 % and the lowest yield obtained from pretreatment method with soaked in water pH7 of 0.08 %.	[29]

extraction method is 5.95 times greater than with microwave hydrodistillation method. This is also supported by previous research which has been done by Kusuma and Mahfud (2016) on patchouli oil extraction with 20 g of dry patchouli leaf material using microwave hydrodistillation and solvent-free microwave extraction method[17]. From the research it can be seen that the yield of patchouli oil for solvent-free microwave extraction method higher with shorter time (2.374% for 45 min) when compared with microwave hydrodistillation method (2.177% for 66 min).

The length of time and the smaller yield obtained using microwave hydrodistillation method is due to the extraction mechanism of microwave hydrodistillation method is in part caused by heat transfer from inside the material to the outside material (Figure 8 (C)) and is also largely due to external heat transfer fromoutside the material into the inside material (Figure 8 (A)). While on solvent-free microwave extraction method, heat transfer occurs mostly from inside the material to the outside material and a small part occurs from outside the material into the inside material (Figure 8 (B)). Based on Figure 8 it can also be seen that there is a combination of two

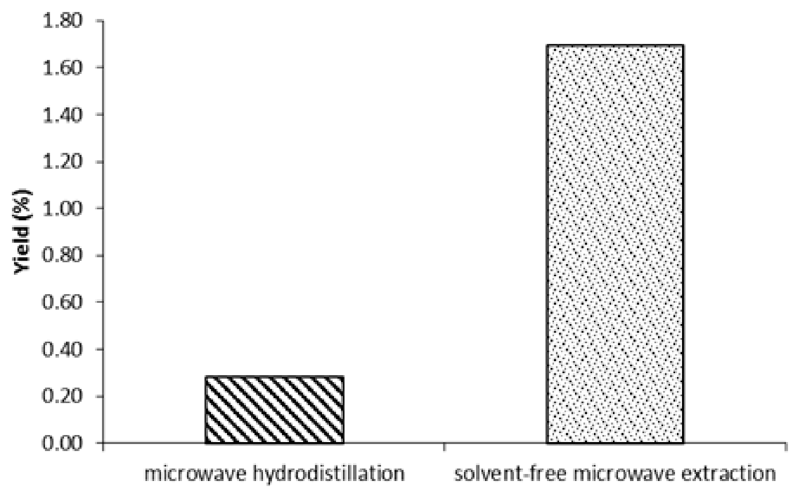


Figure 7. Comparison of agarwood oil yield obtained by using microwave hydrodistillation (powder size, F/S ratio of 0.2 g/mL, microwave power of 300 W, and time of 12 h) and solvent-free microwave extraction method (powder size, F/D ratio of 0.01 g/mL, microwave power of 300 W, and time of 6 h).

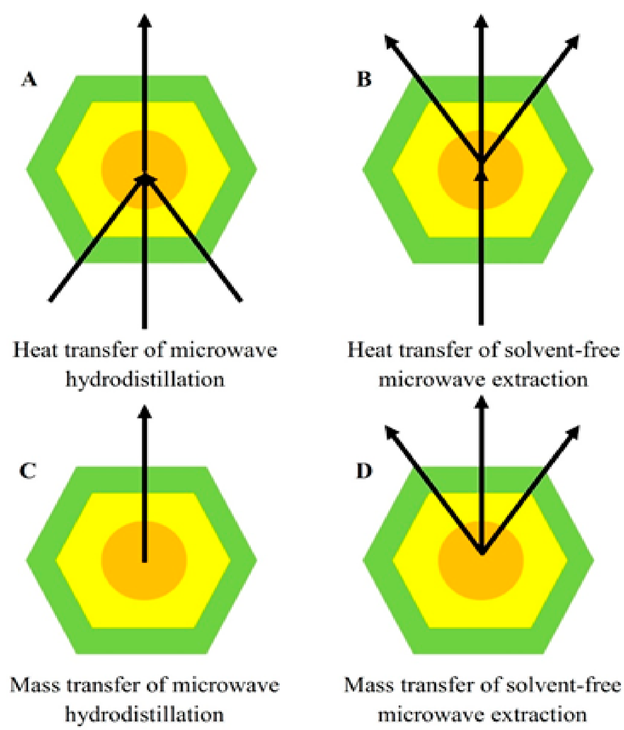


Figure 8. Scheme of mass and heat transfer in microwave hydrodistillation and solvent-free microwave extraction methods.

phenomena (mass and heat) transfer in the same direction that is from inside to outside on solvent-free microwave extraction method. As for the method of microwave hydrodistillation heat transfer and mass transfer goes in different directions and tend to be opposite. It is then possible to explain the yield of extraction using solvent-free microwave extraction method can be higher with a shorter time when compared with microwave hydrodistillation method. In addition, significant internal heating of solvent-free microwave extraction method can also lead to higher internal pressures characterized by the breakdown of oil glands from the extracted material [18].

Based on this research and the results of previous studies it can be seen that solvent-free microwave extraction method is the best method to produce higher yield with shorter time. This can be seen in Table 2.

In the agarwood oil extraction using solvent-free microwave extraction method takes relatively faster than microwave hydrodistillation method. This extraction time is related to the cost and energy required. So to know it, we conducted an analysis of electric consumption and the environmental impact of agarwood oil extraction using solvent-free microwave extraction method and microwave hydrodistillation method. An analysis of electric consumption and the environmental impact of agarwood oil extraction with both methods can be seen in Figure 9.

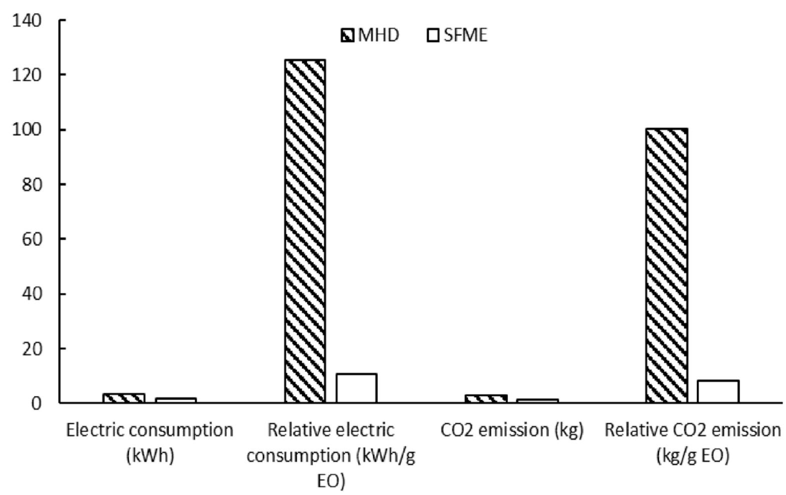


Figure 9. The electric consumption and environmental impact (CO_2 emissions) for the extraction of agarwood oil using microwave hydrodistillation (ratio of feed to solvent of 0.2 g/mL, time of 12 h) and solvent-free microwave extraction method (ratio of feed to distiller of 0.01 gmL^{-1}, time of 6 h) at the microwave power of 300 W.

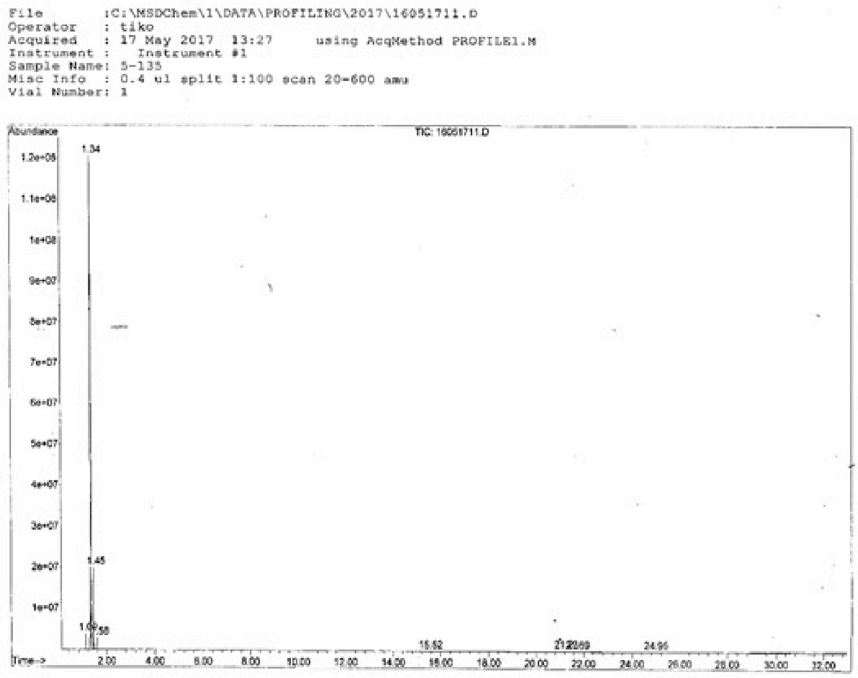
In this study, the electric consumption requirements for agarwood oil extraction by microwave hydrodistillation method was 3.6 kWh, while for solvent-free microwave extraction method was 0.9 kWh. So it can be said that using microwave hydrodistillation method requires 2 times more electricity consumption than solvent-free microwave extraction method. The electric consumption to obtain 1 g of agarwood oil from extraction using microwave hydrodistillation method was equal to 125.4335 kWh, while for solvent-free microwave extraction method was 10.5696 kWh. So it can also be said that microwave hydrodistillation method requires an electric consumption that is 11.8676 times higher when compared with solvent-free microwave extraction method. Thus it can be assumed that the extraction of agarwood oil using solvent-free microwave extraction method reduces energy consumption andrequires lower operating cost when compared with microwave hydrodistillation method [19].

The environmental impact of the essential oil extraction can be known from the amount of CO_2 emissions produced. If 1 kWh of energy from coal or fossil fuels, 800 g of CO_2 will be released into the atmosphere during combustion [4]. The CO_2 emissions produced from agarwood oil extraction with microwave hydrodistillation method of 2.8 kg, while solvent-free microwave extraction method was equal to 1.4 kg. It can be said that the extraction of agarwood oil by microwave hydrodistillation method produces a greater amount of CO_2 emissions than solvent-free microwave extraction method. And to obtain 1 g of agarwood oil, CO_2 emissions produced by microwave hydrodistillation and solvent-free microwave extraction methods were 100.3484 and 8.4557 kg. So it can be concluded that agarwood oil extraction using microwave hydrodistillation method produces greater CO_2 emission when compared with by using solvent-free microwave extraction method. The solvent-free microwave extraction method can reduce the environmental burden by producing less amount of CO_2 in the atmosphere [20]. Therefore, the use of solvent-free microwave extraction method for agarwood oil extraction is suitably method as a new green technique.

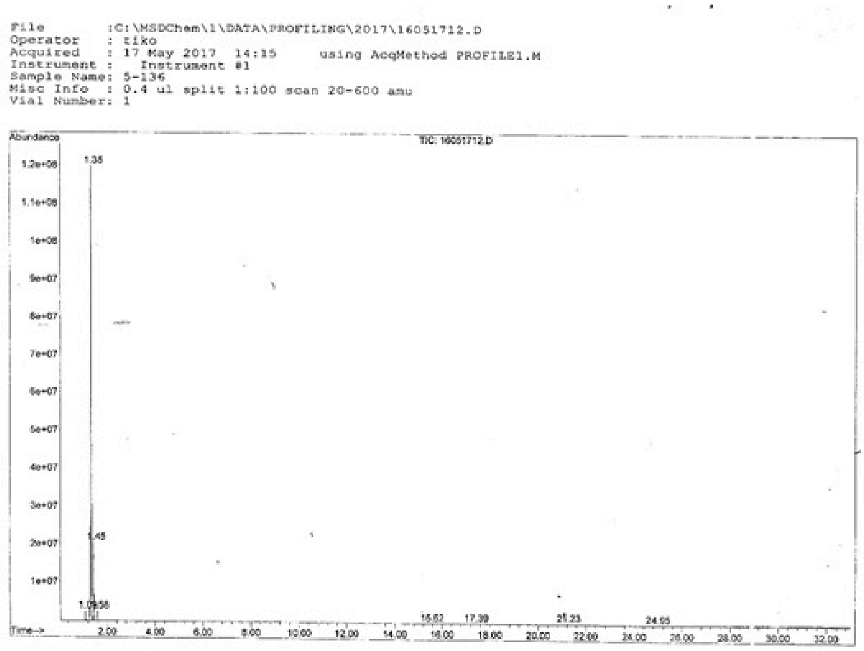
The solvent-free microwave extraction method as a green technique can be developed into a pilot scale in the essential oil refining industry. The use of solvent-free microwave extraction method is more advantageous compared to the conventional methods that already exist related to time and energy use [21]. Where conventional extraction methods that have been used in agarwood oil extraction such as microwave hydrodistillation require time, the solvent and the amount of fuel was quite large. So, the use of solvent-free microwave extraction method in the essential oil refining, in addition to more environmentally friendly also reduces the cost of production [22].

In addition to the yield obtained, the time needed, and the environmental impact, we also need to know the quality of agarwood oil obtained by using microwave hydrodistillation and solvent-free microwave extraction methods. The quality of agarwood oil can be seen based on the results of GC-MS analysis. Based on the analysis, the components contained in agarwood oil obtained using both the microwave hydrodistillationand solvent-free microwave extraction methods were as many as 8 components with the highest component was 2H-Pyran-2-one, tetrahydro-6, 6-dimethyl- for 88.64% using microwave hydrodistillation method and 89.94% using solvent-free microwave extraction method. It can be seen in Figure 10. The component 2H-Pyran-2-one, tetrahydro-6, 6-dimethyl- is an other oxygenated compound group which has more effect on the aroma of essential oils compared to other classes of compounds. Oxygenated compounds have a very fragrant odor and therefore most valuable [23].

Based on the results of GC-MS analysis, the componentscontained in agarwood oil extracted by using microwave hydrodistillation and solvent-free microwave extraction methods only consist of two groups of compounds, namely other compounds and other oxygenated compounds.Where the level of other oxygenated compounds was higher than other compounds. This can occur because of the thermal degradation of microwave, as in the research of Boukroufa et al. (2015) which showed that the Microwave Assisted Extraction (MAE) method can degrade polyphenols from apple pomace[24]. In addition to the MAE method, the extraction of oxygenated compounds was very fast because of the pressure build-up inside the plant material [25]. The number of



(a)



(b)

Figure 10. Chromatogram results of GC-MS analysis of agarwood oil obtained by using (a) microwave hydrodistillationand (b) solvent-free microwave extraction methods.

components classified as oxygenated compounds in agarwood oil extracted by the microwave hydrodistillation method was 90.78%. While other oxygenated compounds in agarwood oil extracted by the solvent-free microwave extraction method was 91.23%. So that it can be seen that the solvent-free microwave extraction method in agarwood oil also has better quality in terms of aroma when compared with the microwave hydrodistillation method.

Conclusion

The extraction of agarwood oil by using solvent-free microwave extraction and microwave hydrodistillation methods is influenced by several parameters such as microwave power, raw material size, feed to distiller ratio used in solvent-free microwave extraction method and feed to solvent ratio in microwave hydrodistillation method. The optimum conditions for the extraction of essential oils from agarwood (*Aquilaria malaccensis*) using solvent-free microwave extraction and microwave hydrodistillation methods are as follows: microwave power of 450 W, F/D ratio of 0.01 g/mL, F/S ratio of 0.4 g / mL, and size of raw material in the form of powder. In this extraction of agarwood oil, solvent-free microwave extraction method is a new green technique which is better than microwave hydrodistillation method. Whereas the solvent-free microwave extraction method that requires faster extraction time (6 hours) obtained a higher yield of 5.95 times compared to using microwave hydrodistillation for 12 hours. In addition, based on the analysis of electric consumption and the environmental impact, the extraction of agarwood oil using solvent-free microwave extraction method requires lower electric consumption and CO_2 emissions production is also lower when compared with microwave hydrodistillation method.So based on this, the use of solvent-free microwave extraction method for agarwood oil extraction has been suitably used as a new green technique. And also, the solvent-free microwave extraction method produces better quality of agarwood oil compared to the microwave hydrodistillation method based on GC-MS analysis.

References

[1] Wong F. Y., Chin S., Perlmutter P. and Marriott P. J., *J. Chromatogr. A*, 2015; **1387**: 104-115. DOI 10.1016/j.chroma.2015.01.096.

[2] Naef R., *Flavour Frag. J.*, 2011; **26(2)**: 73-87. DOI 10.1002/ffj.2034.

[3] Sulaiman N., Idayu M.,Ramlan A., Fashya M., Farahiyah A. and Mailina J., *J. Trop. For. Sci.*, 2015; **27(3)**: 413-419.

[4] Ferhat M. A.,Meklati B.Y., Smadja J. and Chemat F., *J. Chromatogr. A*, 2006; **1112(1-2)**: 121-126.DOI 10.1016/j.chroma.2005.12.030.

[5] Stashenko E.E., Jaramillo B.E. and Martínez J.R., *J. Chromatogr. A*, 2004; **1025(1)**: 93-103. DOI10.1016/j.chroma.2003.10.058.

[6] Golmakani M.-T. and Moayyedi M., *Food Sci. Nutr.*, 2015; **3(6)**: 506-518. DOI10.1002/fsn3.240.

[7] Kusuma H.S., Altway A. and Mahfud M., *J. Ind. Eng. Chem.*, 2018; **58**: 343-348. DOI10.1016/j.jiec.2017.09.047.

[8] Kusuma H.S. and Mahfud M., *J. Appl. Res. Med. Aromat. Plants*, 2017; **4**: 55-65. DOI10.1016/j.jarmap.2016.08.002.

[9] Hu Z., Cai M. and Liang H.H., *Sep. Purif. Technol.*, 2008; **61(3)**: 266-275. DOI 10.1016/j.seppur.2007.10.016.

[10] Chen F., Zu Y. and Yang L., *Sep. Purif. Technol.*, 2015; **154**: 271-280. DOI 10.1016/j.seppur.2015.09.066.

[11] Guenther E., *Essential Oils Volume IVB (in Bahasa Indonesia)*, Jakarta: Universitas Indonesia Press, 1990.

[12] Desai M., Parikh J. and Parikh P.A., *Sep. Purif. Rev.*, 2010; **39(1–2)**: 1-32. DOI 10.1080/15422111003662320.

[13] Spigno G. and De Faveri D.M., *J. Food Eng.*, 2009; **93(2)**: 210-217. DOI 10.1016/j.jfoodeng.2009.01.006.

[14] Metaxas A.C., *Foundations of Electroheat: A Unified Approach*, New York: Wiley, 1996.

[15] Eskilsson C. S. and Bjorklund E., *J. Chromatogr. A*, 2000; 902(06): 227-250.

[16] Guenther E., *Essential Oils Volume I (in Bahasa Indonesia)*, Jakarta: Universitas Indonesia Press, 1987.

[17] Kusuma H.S. and Mahfud M., *RSC Adv.*, 2017; **7**: 1336-1347. DOI 10.1039/C6RA25894H.

[18] Bayramoglu B., Sahin S. and Sumnu G., *J. Food Eng.*, 2008; **88(4)**: 535-540. DOI 10.1016/j.jfoodeng.2008.03.015.

[19] Jacotet-Navarro M., Rombaut N., Deslis S., Fabiano-Tixier A. S., Pierre F. X., Bily A. and Chemat F., *Green Chem.*, 2016; **18(10)**: 3106-3115. DOI 10.1039/c5gc02542g.

[20] Chemat F., Rombaut N., Meullemiestre A., Turk M., Perino S., Fabiano-Tixier A. S. and Abert-Vian M., *Innov. Food Sci. Emerg.*, 2017; **41**: 357-377. DOI 10.1016/j.ifset.2017.04.016.

[21] Petigny L., Périno S., Minuti,M., Visinoni F., Wajsman J. and Chemat F., *Int. J. Mol. Sci.*, 2014; **15(5)**: 7183-7198. DOI10.3390/ijms15057183.

[22] Périno-Issartier S., Zill-e-Huma, Abert-Vian M. and Chemat F., *Food Bioprocess Technol.*, 2011; **4(6)**: 1020-1028. DOI 10.1007/s11947-010-0438-x.

[23] Ferhat M.A., Meklati B.Y. and Chemat F., *Flavour Frag. J.*, 2007; **22**: 494–504. DOI 10.1002/ffj.

[24] Boukroufa M., Boutekedjiret C., Petigny L., Rakotomanomana N. and Chemat F., *Ultrason. Sonochem.*, 2015; **24**: 72-79. DOI 10.1016/j.ultsonch.2014.11.015.