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Improving the quality of Patchouli Oil Using Microwave Distillation

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Abstract : There are so many plants that could produce volatile oil grow in Indonesia. Volatile oil is a compound, mostly liquid, produced from the parts of plants, such as roots, cortex, trunks, leaves, fruits, seeds, or flowers through distillation process. Volatile oil is commonly used as additional aroma in food, soap, tooth paste, and medicine. In order to fulfil these demand, volatile oil is extracted from various kinds of plant as the demand of it is getting higher. One kind of volatile oil is patchouli oil, the most favorable in Indonesia for its high economic value. Patchouli oil has a fresh and distinct fragrance as well as high fixation power that is incomparable to any synthetic product. A new process is developed to improve the quality of patchouli oil, that is by using microwave distillation process. This process is the combination of microwave and distillation system. This study aims to analyze the most suitable distillation time and temperature to produce higher patchouli oil content and rendemen than Indonesian National Standard (SNI) determined. Variable used in this research are 200 watt, 300 watt, 400 watt and distillation time of 40 minutes, 80 minutes, 120 minutes, and 160 minutes. This study resulted patchouli oil content as much as 84.41756% and produced rendemen about 3.4%. Meanwhile the production based on SNI is the minimum standard of patchouli oil content, that is 35%, and commonly the rendemen acquired is 2.5%. Keywords : microwave, patchouli oil, patchouli oil content.

Introduction

Studies performed by Manurung, T. said that volatile oil or also called as essential oil, ethereal oils is a natural extract commodity produced from the leaves, flower, trunk, seeds or even the ovary of plants. There are at least 150 kinds of volatile oil spread out in International market and 40 of them are produced in Indonesia. In Indonesia, there are 40 kinds of volatile oil which are being traded, 12 of them has been well developed and being exported. Not to mention, Indonesian production of volatile oil has dominated the market of volatile oil in International trading, such as patchouli oil (800 tons of volatile production), ylang-ylang (25 tons of volatile oil production), vetiver (30 tons of volatile oil production), lemon grass (500 tons of volatile oil production), nutmeg (350 tons of volatile oil production), and clove (2,500 tons of volatile oil production)⁶.

There are a lot of volatile oil made in Indonesia, yet only a few of them had been developed or is being developed. The industry of volatile oil in Indonesia has been founded since the colonialization era. Unfortunately, there is no any vast improvement in terms of its quality and quantity. This is because most of the volatile oil factories use traditional technology and have limited production capacity³.

Patchouli oil has a clear yellow color and distinct fragrance, the component of Indonesian Patchouli oil are β -patchoulene(2,9-3,8%), α -guaiene(12,1-15,2%), caryophylene(3,3-3,9%), α -patchoulene(5,1-5,9%), α -

bulnesene(4,7-16,8%), *norpatchoulenol*(0,5%), *patchouli alcohol*(32-33,1%) and *pogostol*. Here we can see that the main compotent of Patchouli oil is patchouli alcohol⁴.

Patchouli alcohol is sesquiterpene compound, that is tertiary tricyclics alcohol, not dissolve in water, dissolve inalcohol ether or other organic solvent, the boiling point is 280.37° C and the crystal of it has the melting point on 56° C².

The molecule structure of patchouli oil is depending on several factors, those are the soil condition on where the plants grow, the age of the leaves, cutting technique, drying process, processing techniques, acidity and plants' variety⁸.

Patchouli oil can be combined with other etheric oil, it dissolves in alcohol and not easy to evaporate. For these characteristics, patchouli oil is used as fixative or to bind other fragrance substances. Thus, patchouli oil has an essential role in perfumery¹¹.

Distillation is the process to transform liquid to vapor, and the vapor would be converted back to liquid. Distillation operation unit is the method used in sorting the components during the vapor phase and liquid phase. Vapor phase is formed from liquid phase through evaporation process in its boiling point. First condition in the sorting operation of the components through distillation is the composition of vapor should be different from the composition of liquid with the balance of all the solutions, and all the components are evaporated. The temperature when the liquid boils is its boiling point in the atmosphere pressure used¹.

Several ways to improve the quality of patchouli oil are the cultivation process of patchouli plants, distillation process and the equipment used, the treatment upon the ingredients, patchouli oil purification process, and the packaging. There are several methods to separate or produce patchouli oil, those are distillation, extraction, etc. But the most common system used is distillation. Based on the distillation technique used, steam-hydro distillation could produce better rendemen than conventional method, that is water distillation⁵.

Optimal operation and equipment design are also important to produce fine quality oil. Yet, this method takes pretty long time to produce good patchouli oil. Therefore, a new method like microwave distillation is important to be developed in order to make distillation process faster⁷.

Previous Studies

- The research by Bambang Setyoko under the title of determining optimal operation condition in patchouli oil distillation. This research varied the vacuum pressure in producing the oil, start from -30cmHg to -70 cm Hg. From this research, the condition that allowed higher production of oil is 50 cmHg pressure and temperature of 80°C that resulted 2.5% rendemen and 28% patchouli content. This result has fulfilled the criteria to be exported overseas. After harvesting patchouli leaves, the leaved then dried in glass or fiber house. It may not have direct contact with sun rays as it could reduce the content of its volatile oil¹⁰.
- 2. The research by Setya,H., Budiarti,A., Mahfud,MDP method needs 60-120 minutes to produce maximum amount of rendemen, while MDS needs 120-140 minutes. Microwave distillation system produce more oil rendemen in shorter period of time compare to conventional method of distillation which requires 6-8 hours process⁹.

Methodology

A. The materials needed

Patchouli leaves that is used is in fresh/wet condition, aged for about 6 months from Dampit, Malang Region. Then the leaves are dried for 3-4 days. Before it is distillated, the leaves are chopped first.

B. Description of the tools

A number of tools that are used for the microwave distillation method with the steam are as follow:

1. One unit of Microwave that used as the heater. With the dimension of 50 cm long, 40 cm wide and 40 and 40 cm tall. As well as output power that used as high as 400 W with the frequency of 2450 MHz

- 2. Distiller used is in a shape of double-necked flask made from glass with the volume of 1000ml and a connector made from glass used for connecting the distiller and the condenser.
- 3. A steam generator consisted of glass made flask with 1000 ml in volume and a hot plate used to heat the water into a steam.
- 4. The condenser used is the *liebig* condenser functioned to cool down the steam that turns it into liquid.
- 5. The separator used to separate Patchouli oil and the water.
- 6. Temperature measurer such as thermometer that is used to measure the temperature on the steam generator and thermocouple to find out the temperature in the microwave.

The arrangement of the tools on microwave distillation method with the steam provided completely on Picture 1.



Picture 1. Scheme of tools on Microwave Distillation Method with Steam

Description:

- 1. Double-necked flask
- 2. Heat controller
- 3. Timer
- 4. Liebeg Condenser
- 5. Separator funnel
- 6. Erlenmeyer
- 7. Thermocouple
- 8. Hot plate
- 9. Temperature controller
- **10.** Thermometer
- 11. Flask (1000 ml)

C. Procedure

For the microwave distillation method by using steam has used the procedure as follow, began by weighing the Patchouli as much as 50 gram. Then inserting the weighed leaves on the distillation flask by adding a little water in the distillation microwave. Then heating the water in the flask to then used as the steam generator, the heating proses uses the hot plate. Igniting the microwave heater and setting the power of the microwave according to the variable. Then stop the process according to the variable that determined.

Containing distillate in the separator funnel and separating the oil from the water with it, then contain the oil in the reaction tube. Then taking water free oil and finally analyze the oils produced.

D. Condition of the Operation and the Variable

- Atmospheric pressure
- Mass of the patchouli leaf : 50 gram
- Rate of water in the leaf : 14%-17%
- Temperature of the distillation: 105, 110, 115, 120° C
- Size of the leaf ± 2 cm
- Time of observation : 40, 80, 120, 160, 200 minutes.
- Power for the heating : 200, 300, 400 watt.

Result and Analysis

The result gained needs to be compared with the requirements of National Standard of Indonesia, where the quality of Patchouli oil are as follows :

Type of Test	Unit of	Requirement
	Measurement	
Color	-	Bright yellow – reddish brown
Density 25 [°] C	-a	0.950 - 0.975
Refraction Index (nD ²⁰)	-	1,507 – 1,515
Solubility in Ethanol 90% on	-	The solution is clear or has the light opalescence in
$20^{\circ}\mathrm{C} \pm 3^{\circ}\mathrm{C}$		comparison of volume 1:10
Number of Acidity	-	Max. at 8
Number of Ester	-	Max. at 20
Optical Rotation	-	(-)480 – (-) 65
Patchouli alcohol (C ₁₅ H ₂₆ O)	%	Min. 30
Alpha Copaene ($C_{15}H_{26}$)	%	Max. 0.5
Content of Iron (Fe)	Mg/kg	Max. 25

Table 5.1 the Requirements of Patchouli oil quality of SNI 06-2385-2006

1. The result of analysis using GC, for the Patchouli that is exposed to wind and are chopped with the distillation tool completed with the microwave, frequency of 2450 MHz on 200 watt, is as follow :

Table 5.2 : Result of observation on Patchouli oil for 200 watt

Distillation time	Yield (%)	Rate of Patchouli oil	Refraction Index
40	1	64.16174	1.509
80	1	51,69519	1.505
120	1.76	55.38630	1.505
160	.176	49.43968	1.508
200	3.4	84.41756	1.508

Table 5.3 : Result of observation on Patchouli oil for 200 watt

Distillation time	Density	Number of Acidity	Number of Ester
40	0.874	19.2562	14.2773
80	0.944	17.8283	11.8855
120	0.922	18.2537	14.2687
160	0.876	19.2123	13.2123
200	0.984	17.1036	13.1836

2. The result of analysis using GC, for the Patchouli that is exposed to wind and are chopped with the distillation tool completed with the microwave, frequency of 2450 MHz on 300 watt, is as follow :

Distillation time	Yield (%)	Rate of Patchouli oil	Refraction Index
40	0.64	47.30732	1.508
80	1.78	57.58026	1.507
120	2.16	53.43796	1.506
160	2.04	74.80910	1.508
200	2.92	93.50092	1.507

Table 5.4 : : Result of observation on Patchouli oil for 300 watt

Table 5.5 : Result of observation on Patchouli oil for 300 watt

Distillation	Density	Number of Acidity	Number of Ester
time			
40	1.048	21.4122	16.0592
80	0.906	24.7682	18.5762
120	0.864	19.4791	13.4792
160	0.848	19.8466	14.8467
200	0.874	19.2563	14.2603

3. The result he distillation tool

of analysis using GC, for the Patchouli that is exposed to wind and are chopped with the distillation tool completed with the microwave, frequency of 2450 MHz on 400 watt, is as follow :

Table 5.6 : Result of observation on Patchouli oil for 400 watt

Distillation time	Yield (%)	Rate of Patchouli oil	Refraction Index
40	0.96	43.52550	1.505
80	2.2	71.09464	1.505
120	2.8	39.21643	1.507
160	2.04	80.34757	1.508
200	0.76	30.02081	1.332

Table 5.7 : Result of observation on Patchouli oil for 400 watt

Distillation time	Density	Number of Acidity	Number of Ester
40	0.940	23.8723	17.9043
80	0.856	19.6612	13.1075
120	0.884	19.0384	12.6924
160	0.990	17.000	14.8023
200	0.900	18.7000	13.7142

Analysis

If we observe from the Table 5.2 until 5.7 that is seen from the requirement of the quality of Patchouli oil SNI 06-2385-2006 are as follow:

- 1. For the heating power of 200 watt, it is gained that the yield 1 3.4% of Patchouli rate of 49.43968 until 84.41756%, this is far better than what is required by the SNI that is above 30. The refraction index has two values that is not meeting the requirement that are between 1.507 1.515. On the density made, the lowest score is 0.874 and the highest is 0.984 which is less suitable since the value is below the one allowed which between 0.95 0.975. the number of acidity also not quite match with the requirement which is above the one allowed which is maximum at 8, but the result is above 17. The number of Ester is good, which is fulfilling the requirement, resulted from 11.8855 14.2773 which means still allowed that is below 20.
- 2. On the 300 watt power, the yield made is from 0.64 292, the rate of good Patchouli oil starts from 47.30732 93.50092% and that is above the requirement of SNI 30, the refraction index ranging from

1.506 - 1.508, one point that is not quite close to the one allowed from the five points that is being analyzed. The density that is produced 0.848 - 1.048, some points are less suitable with the requirement that is 0.950 - 0.975. the acidity number is not suitable with the SNI (maximum at grade 8), the result is above 19. The ester number still meets the requirement, which is still below 20, which is at maximum is 18.5762.

3. While for the 400 watt, the yields made are good. Three points are above 2 percent, which the highest is 2,8%, for the rate of Patchouli alcohol is really good with peak score of 80.34757%, the Refraction index of two points close to the one allowed. For the density on the distillation time of 160 minutes fulfill the requirement of 0.99, the value of acidity number 5 in points still less match the requirement of above 8, while all the ester number fulfill the requirement of above 20.

Conclusion

From the research of chopped Patchouli leaves that exposed to the wind using the distillation tools of microwave with the frequency if 2450 MHz creates conclusions as follow :

- 1. By using the tools that modified, the best results is gained from the distillation time of 200 minutes, with a heating power of 200 watt, produce high levels of patchouli oil 84.41756.% It's nicer than the SNI is a maximum of 30%.
- 2. By using the tools that modified, the best result is gained from the distillation time of 200 minutes, with the heating power of 200 watt, produce the yield of 3.4%. that usually creates only yield 2.5%.
- 3. By using the modified tools, the best result is gained from the distillation time of 200 minutes, with the heating power of 200 watt, creates the refraction index of 1.508, the density of 0.984, acidity number 17.1036, ester number of 13.1836.

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