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Determinating Patchouli Alcohol of Patchouli Oil Using Distillation Technique

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Abstract: Essential oil, also known as ethereal oil or volatile oil is widely needed in daily life. A kind of essential oil is patchouli oil. In the international trade market, patchouli is traded in the form of oil and known as "patchouli oil". Amongst various essential oils in Indonesia, patchouli oil is being a belle. Every year, more than 45% of foreign exchange gained from essential oils is from patchouli oil. In the term of patchouli oil, Indonesia plays a quiet significant role, approximately 90% of world's demands for patchouli oil is met by Indonesia. This study was aimed at knowing how to treat the material and find out the appropriate distillation pressure to generate patchouli oil containing high patchouli alcohol and high yield. The method used was steam distillation which spent 6 hours long, with the operational pressure of 0,1; 0,2; 0,3; 0,4; 0,5 kg/cm^2, using patchouli leaves with three treatments are fresh leaves, aerated leaves and burned leaves in the oven. This study's results concluded that: the best result was obtained from the burned leaves in the oven where the pressure was 0.4 kg/cm^2, yield 2%, containing patchouli alcohol 40.06% and specific gravity was 0.961. The treatment to materials and stem pressure are not significantly affecting the specific gravity of essential oil. The organoleptic test results showed that it produced various colors from light yellow until tawny and all of them have the typical smell of patchouli oil.
Keywords : Patchouli alcohol, Steam Distillation, Yield.

Introduction

Essential oil, also known as ethereal oil or volatile oil is widely needed in daily life. Such oil is used to be material of fragrance in foods, soaps, tooth pastes, fragrances and drugs. To meet those needs, most of essential oil supplies are taken from various plants which are producing essential oil since its utilization in human life is increasing. In Indonesia, there are about 40 kinds of plants producing essential oil but until today, there are only 12 kinds of plants have been developed are vetiver, citronella, nutmeg, clove, eucalyptus, patchouli, ylang, pepper, ginger, sandalwood, addle and mesoyi. Amongst such 12 kinds of plants, there are 9 of them have penetrated the export market including patchouli. In international trade market, patchouli is traded in the form of oil and known as 'patchouli oil'. Patchouli oil consists of compounding terpene with alcohols. While aldehyde and esthers give typical smell such as patchouli alcohol. Patchouli alcohol is a compound which determines the smell of patchouli oil and being the greatest component[5].

The components 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*. From these composition, can be seen that the main component of patchouli oil is patchouli alcohol.

The hexane extract was further separated and purified to obtain patchouli alcohol (0.05% dry wt), a mixture of *β*-sitosterol and stigmasterol (0.09% dry wt) and 7,3',4-tri-*O*-methyleriodictyol (0.04% dry wt). Antibacterial activity assay showed that patchouli oil could inhibit *Staphylococcus aureus* and *Bacillus subtilis* better than the hexane extract[6].

Previous Studies

Studies performed by Niken Harimurti, Tatang H Soerawidjaja, Djajeng Sumangat and Risfaheri titled Patchouli Oil Extraction (*pogostemon cablin* benth) using hydrodifussion technique under the pressure of 1 – 3 bars. In general, patchouli oil extraction in Indonesia is carried out under atmospheric pressure, while in this study, is under higher pressure between 1 – 3 bars. This study aimed at optimizing the conditions of operating process so as to obtain a belle quality of patchouli oil. Every year, more than 45% of foreign exchanges are generated from essential oil of patchouli oil[1].

Table 1 Patchouli oil quality requirements SNI 06-2385-2006

Test type	Conditions
Colour	Light yellow - reddish brown
Specific Gravity 25°C	0,950 – 0,975
Refractive Index (nD^{20})	1,507 – 1, 515
Solubility in ethanol 90% at the temperature 20°C ±3°C	Diaphanous solution or light opalescence with volume ratio of 1:10
Acid Number	Max. 8
Esther Number	Max. 20
Optical rotation	(-) 48° – (-) 65°
Patchouli alcohol ($C_{15}H_{26}O$)	Min. 30 %
Alpha copaene ($C_{15}H_{26}$)	Max. 0,5 %
Iron content (Fe)	Max. 25 mg/kg

In the context of patchouli oil product, Indonesia plays a significant role, about 90% of world demand met by Indonesia. The important quality parameter of patchouli oil is patchouli alcohol content standardized at minimally 30% in Indonesia. The higher patchouli alcohol content, the better oil quality since the compound of patchouli alcohol determines the fragrance level of the oil produced. Nowadays, patchouli oil produced from patchouli distillation (*Pogostemon Cablin Benth*) is being developed in Jawa and Sumatera. However, patchouli alcohol contained in Aceh patchouli oil in Jawa (PA ≤ 30%) is lower than it in Sumatera (PA ≥ 30%). The quality of patchouli oil is affected by several factors such as leaves quality, distillation method and storage duration. The distillation method includes initial harvesting process to the final process of producing essential oil. According to Sunardi, a drying system can increase the yield of patchouli oil produced. This is supported by a study by Noviar, 2010 who found a combination of treatments by sunning for 5 hours and letting it exposed to the wind for 3-4 days. After the drying process, it is necessary to chop up. The chopping before distillation process may also increase the oil yield. Sufficient quantity and quality in accordance with the applicable standard. The variables of pressure and time give significant effect on the parameter of patchouli oil yield. The findings show that the best product was resulted by extracting under the pressure of 3 bars over 3 hours (which produced yield of 0.04% V/W and the density 0.9739 kg/m^3)[2]. All modified process must be performed to produce the best results. The experiment of isolation patchouly alcohol from patchouly oil was conducted using vacum fractio distillation method. The isolated patchouly alcohol was identified by gas chromatography-mass spectrometry met-hod. The isolation gave 1% yield of pat-chouly alcohol, and the purity of isolated patchouly alcohol was 91.5%[7].

The main purpose of this research is to improve the quality of patchouli oil by the purification process technologies after oil refining process. In this research the purification of the dirty and dark oil can be carried out using complexometry method where the iron metals are attached by chelating agent chemical to form the complex compound. The purification experiment was carried out to evaluate the influence of chelating agents (citrate acid, tartarate acid and EDTA) their concentration and duration of mixing on the quality of pure oil produced. Material used was the crude patchouli oil from the small distilling industry in north aceh. The experiment used a completely randomized design, arranged factorially with three replications. The results

showed that the purification process can improve the oil quality, especially in terms of color, physicochemical properties and concentration of its main components. From the oil refining process can produce a brighter and the characteristic also meet the quality requirements of national standards[3].

A study conducted by Nur Hidayat, Desi Wiwis Sahendrati and Nimas Mayang Sabrina Sunyoto (2012), which was entitled 'The effect of the Packaging Types of Dry Patchouli-Leaves (*pogestemon cablin* benth.) and the Length of Distillation Delay on the yield of Patchouli Oil'. This study was aimed at recognizing the effect of the packaging types of dry patchouli-leaves and the length of distillation delay on the yield of patchouli oil. From this study, it can be concluded that the delay of distillation process is affecting the level of water, total fungi and total bacteria rather than types of packaging. The packaging types of dry patchouli-leaves and the length of distillation are not affecting significantly the randemen of patcouli oil, while the best treatment was found in the leaves' storage treatment performed by storaging the leaves in gunny sacks and delaying the distillation for 5 days[4].

Study performed Bambang Setyoko (2007) with the title 'Determining the optimal operating conditions in the distillation of patchouli oil. Such study had varied vacuum pressures at the time of taking the oil from – 30 cmHg to – 70 cmHg. The results showed that the condition of distillation to produce the best patchouli oil was at the pressure of – 50 cmHg and the temperature of 80^{o}C the yield of 2.5% and the content of patchouli oil 28 % so as to meet the terms of standards if it will be exported to foreing countries. After being harvested, the leaves were dried up in glasshouse of fiber house and should not dry under the sun directly that reduces the content of essential oil[11].

A study conducted by Heni Setiyowati and Noor Fitri entitled Increasing the Quality of Patchouli Oil by Modifying the pH-level of Distillation Water. The distillation of patchouli oil using waters at the pH of 7, 9, 10 and 12 generated various weights of yield. The maximum weight of yield was produced by using water with the pH of 9. In determining specific gravity results, can be seen that the higher the pH level of water used, the higher the specific gravity. At the pH of 12, the yield was at the minimum value as the more of substances dissolved in the distillation water slow down the steaming process. These made yield weight differences though in similar durations. The analysis using GC-MS had found compounds contained in patchouli essential oil. The findings indicated that the water with modified pH of 7 and 9 produce patchouli alcohol of 34.5 and 35.62, while of 10 and 12 produce 56.72 and 58.35. The higher pH may neutralize organic acids in the ground water so as to obtain the better result than the water with no additional alkaline level[9].

A study performed by Novita Setya H, Aprilia Budiarti and Mahfud (2012), entitled the Process of Essential Oil Extraction from Pathouli Leaves using Microwave. This method was almost similar with the steam distillation method. Nevertheless, the simplicia in this method are soaked and exposed to the microwaves to increase the yield and accelerate the distillation duration. The conclusions we got from this study of patchouli oil extraction using the method of microwave distillation are as follows:

1. The usage of *microwave distillation* may increase the yield within a shorter time than conventional method.

2. From the study performed, it is found that the percentage of oil yield produced by using MDP is more than it by using MDS for intact and chopped patchouli leaves (±2cm) ranging from 1.3567 – 2.4566, while the percentage of yield for MDS is ranging from 1.4604-1.9485.

3. The treatment to the chopped leaves (±2cm) considered better than in quantity than the treatment to the intact leaves. This has been proven from the percentage of oil yield generated from the chopped leaves (±2cm) which of 2.4566% for MDP method 1,9485 % for MDS

4. The higher the warming temperature, the greater the volume of oil generated, so that the percentage of yield produced will also increase by the increase of temperature Thus % yield generated will also increase as more and magnitude of warming temperatures.

5. The qualitative analysis results (GCMS) showed that the largest component of patchouli oil from it leaves is patchouli alcohol where its percentage in chopped patchouli-leaves is higher (31.88%) than it in intact leaves (27.24%).

6. To produce maximum yield, it needs 60 – 120 minutes long using MDP and 120 – 140 minutes using MDS. With similar mass and yield, microwave distillation method can increase oil yield faster than conventional distillation method which need 6 – 8 hours[10].

A study performed by Nurchayati, Yulita and Setiari, Nintya (2005) The Content of Essential Oil of Patchouli Leaves (Pogestemon eablin Benth) at Different Temperature and Drying Duration, explained that the optimal temperature of burning patchouli leaves in the oven to produce high yield is at 50^0C over 3 hours[8].

Distillation can be defined as the separation of components of a mixture of who liquid materials or more based on the boiling point of each of these substances. The process of essential oil distillation is divided into three, are:

a. Distillation with Water

In this method, the material which will be distilled will directly contact with boiling water. Such material is floating in water or completely submerged depending on its specific gravity and amount to distillation. The water is heated by the usual heating method; by direct heat, steam coat, closed circular steam pipes, or opened or perforated circular steam pipe. The typical characteristic of this method is the direct contact between the materials and the boiling water. This distillation method is appropriate for dry simplicia which are not damaged by boiling condition. The advantages of this method is that the tools are simple, easy to get and easy to use. While the disadvantages are that not all materials can be treat using this method, especially materials containing soap fractions, materials soluble in water and easily burned materials. In addition to that, the existence of water may cause hydrolysis and prolong distillation duration.

b. Distillation by Water and Steam

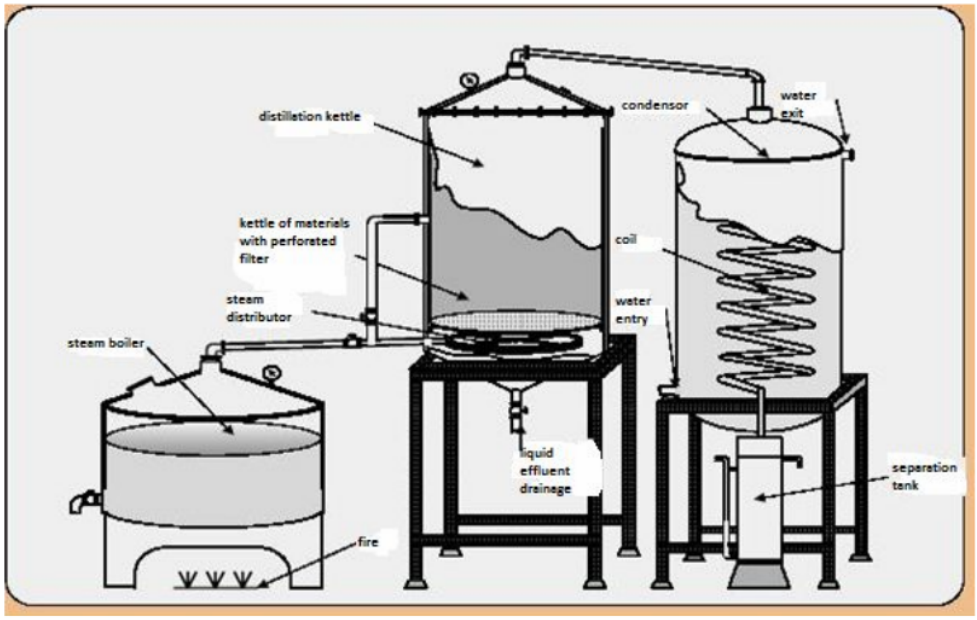
In this distillation method, all materials are put on the shelves or perforated filter. The distillation kettle is filled by water until the water surface level is not far below the filter. The water can be heated by various ways or by wet saturated steam with low pressure. The typical characteristics of this method are, first, the steam is always on wet, saturated, and no too hot conditions. Second, the materials are contacting only with the steam not with the water. The advantages of this method are that the tools are easy to get and the hydrolysis is hardly to occure, so that the quality of oil produced is quite good. While the disadvantages are, amongs other, that only oil which has boiling point lower than water's point can be distilled. It makes the distillation results will not perfect.

c. Distillation with Steam

Basically, this is similar to the previous method, but the water is not filled to the equipment. The steam used is saturated steam or overheated steam under the pressure of more than 1 atm. The steam is flowed through porous circular steam pipe under the materials, while the steam is moving upward exposing the materials on the filter. This method is well-used to distilling essential oil from seeds, roots, or woods that are generally containing oils with high boiling points. The advantages of this method are that the pressure is can be set, the duration is short, not hydrolysis occurs and the quality of oil produced is quiet good.

Experimental

Equipments used



The Materials used:

- Patchouli leaves and plant (*pogestemon calbin bent*)
- n-hexan
- nitrogen gas (N_2)
- water

Research Procedure.

1. Preparation stage
   - Choosing good leaves
   - Conducting 3 treatments :
     - Fresh : fresh patchouli leaves without any drying process
     - Withering : the leaves are exposed to winds for 3 – 4 days at room temperature.
   - Drying : the leaves are burned in the oven at the temperature of 50^0C over 4 hours
   - Varying the leaves based on the treatments (fresh, withering, drying with oven)
   - Weighing the leaves will be distilled
   - Chopping the leaves to the smaller size (randomly)

2. Distillation stage
   - Entering 1 kg of leaves or materials into the distillation equipment.
   - Entering the water and heating the boiler.
   - Setting the steam of the boiler in accordance with appropriate variable.
   - Performing the distillation for 6 hours.
   - Collecting the distillate in erlenmeyer.
3. Stage of oil and water separation.
   - The oil which still contain water is added by 15 mL of n-hexane.
   - The water and n-hexane are separated using separating funnel.
   - The water resulted from the separation then be added by 10 mL of n-hexane (flushing)
   - The n-hexan of flushing I and flushing II are mixed
   - The mixture of n-hexane is steaming in the operating temperature condition 40°C, 350 mbar
   - The steaming result is flowed by nitrogen gas.
   - Repeating the procedures of the preparation stage until the distillation stage with variables predefined before.

Results and Discussion

Table 2. Organoleptic-test results

Leaves condition	Pressure (kg/cm^2)	Colour	Smell
Fresh	0,1	+	Less strong
Fresh	0,2	+	Less strong
Fresh	0,3	+	Less strong
Fresh	0,4	+	Less strong
Fresh	0,5	+	Less strong
Withering	0.1	++	Patchouli Typical smell
Withering	0.2	++	Patchouli Typical smell
Withering	0.3	+++	Patchouli Typical smell
Withering	0.4	+++	Patchouli Typical smell
Withering	0.5	+++	Patchouli Typical smell
Oven	0.1	++	Patchouli Typical smell
Oven	0.2	++	Patchouli Typical smell
Oven	0.3	+++	Patchouli Typical smell
Oven	0.4	+++	Patchouli Typical smell
Oven	0.5	+	Patchouli Typical smell

These finding were tested by organoleptic-test and resulting in as can be seen on the Table 2. The smell of patchouli oil produced by the fresh leaves is less strong, while the smell produced by withering and burning in the oven are typically. About the colour, the leaves coloured light yellow are mostly found from the fresh leaves, while from the withering and the oven are more brownish. Those are in accordance with SNI standards; light yellow – reddish brown.

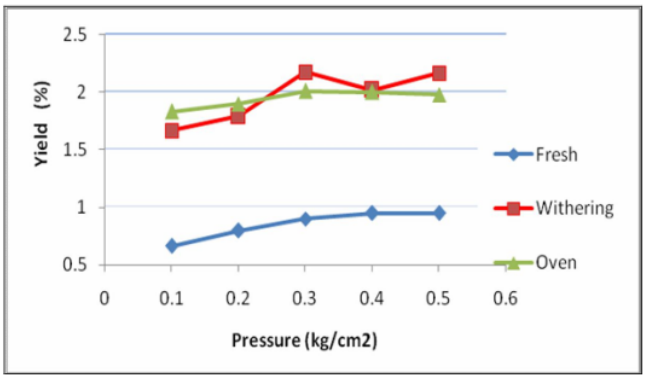


Figure 1 The relationship between yield and pressure on the treatment to patchouli leaves

From Figure 1 can be seen that the fresh leaves treatment always increases the yield, in line with the increase of pressure 0.664 to 0.95. It was caused by the thickness of cell walls and the much water contained, so that the cells are more difficult to penetrate by the steam, the water inside the cell is more dominant and evaporating first before the oil which contained on the cell walls. After the water in the cell cavity is completely evaporating, the steam then excrets water and oil contained by cell. These make the duration of distillation process longer. The distillation of fresh leaves produces low yield, the cells containing oil are just a little on the leaf surfaces and most of them are inside the leaves.

While the withering is resulting in patchouli oil more than the other treatments (fresh and oven). The higher yield is produced in withering condition that is 2.172% where the water is very little and making the leaf pores open and the oil exits. The result of withering under the pressure of 0.1 kg/cm^2 to 0.3 kg/cm^2 was increasing stably of 1.664; 1.788 to 2.172% but under the pressure of 0.4 kg/cm^2 was decreased and resulting 2.016% and under the pressure of 0.5 kg/cm^2 was increasing once more though the increase was not as high as under the pressure of 0.3 kg/cm2 by 2.164 though the difference was only 0.008%.

The result of oven treatment is high enough though lower than the previous treatment. Under the pressure of 0.1 kg/cm^2 to 0.3 kg/cm^2 there was an increase of 1.828% ; 1.898% and 2.008% but under the pressure of 0.4 kg/cm^2 there was a decrease to 2,000% and under the pressure of 0.5 kg/cm^2 there was second decrease to 1.978% though the fluctuating was not too high. From the resuts, it can be estimated that during the process in the oven, several % of patchouli oil were steaming and the distillation equipment was not proper, the leaves were too dry so that the steam was so difficult to penetrate the leaf cells.

The data indicates that there were results which are not good though there were several treatments better than production of farmers in Balaisari Village of 1.3%. The yield condition of burned leaves is higher than it of fresh leaves since the drying process conducted is affecting water reduction in the cells so that the cells are easily to be penetrated by the steam. Since the water contained is less, the steam is easier to evaporate the oil. According to the statement of Hobir et al, (2003)[12] perfoming withering first will increase production of rendemen as the internal cells are easy for the steam to penetrate the internal cells during the distillation.

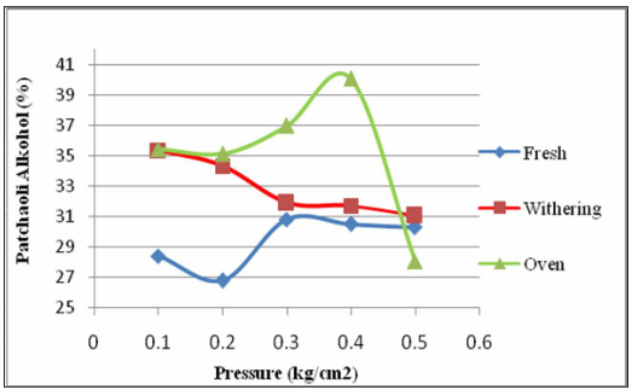


Figure 2. The relationship between patchouli alcohol and pressure on the treatment to patchouli leaves

Figure 2 shows that the higher level of patchouli alcohol was in the oven treatment under the pressure of 0.4 kg/cm^2 that was 40.06% and the lower one was in fresh leaves condition under the pressure of 0.2 kg/cm^2 that is 26.8%. It might be happened when the fresh leaves were still containing water so that the steam were difficult to open the leaf cells and the steaming component was another component of patchouli oil so thath the patchouli alcohol under the low steam pressure also generated low patchouli alcohol.

The result of patchouli-alcohol-content test shows that all treatments met the standard of SNI (min. 30%) where for the fresh leaves was 29.37, for withering leaves was 32.88 and for oven leaves was 35.13. That patchouli alcohol content may be affected by some factors such as the storage. The storage of this study was considered not good because it was using glass bottle at uncontrolled temperature. It can reduce the existing patchouli alcohol which was more than patchouli alcohol produced in the study of Setya N[13]. entitled Extracting Essential Oil of Patchouli Leaves using Microwave) at 31.88%.

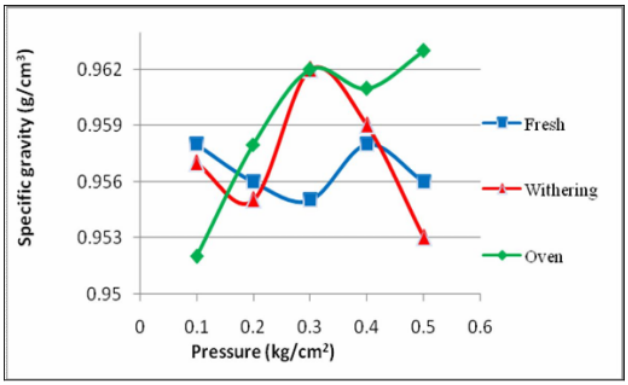


Figure 3. The relationship between the specific gravity and the pressure on the treatment to patchouli leaves

Figure 3 explains that the lowest specific gravity was in the oven treatment under the pressure of 0.1 kg/cm^2 that was 0.952 g/cm^3 while the higest one was also in the oven treatment under the pressure of 0.5 kg/cm^2 that was 0.963 g/cm^3. The steam pressure (0.1 kg/cm^2 to 0,5 kg/cm^2) was not significantly affecting the specific gravity resulted. This specific gravity was affected by the percentage of the components of patchouli oil and water contained in the oil. All data obtained were in the range of SNI from 0.950 – 0.975 g/cm^3.

Conclusions

1. The best result was obtained using oven treatment under the pressure of 0.4 kg/cm^2, yield of 2,00%, patchouli alcohol of 40,06% and specific gravity of 0.961.
2. Material treatments steam pressure was not significantly influencing the specific gravity of patchouli oil.
3. The organoleptic-test resulted in light yellow to tawny colours and all of them had the typical smell of patchouli oil.

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