Identification of patchouli oil chemical components as the results on distillation using GC-MS

D Ermaya 1*, S P Sari 2, A Patria 2, F Hidayat 1, F Razi 3

1Faculty of Agriculture Technology, Serambi Mekah University, Indonesia
2Faculty of Agriculture, Universitas Syiah Kuala, Indonesia
3Faculty of Engineering, Universitas Syiah Kuala, Indonesia

*E-mail: dewi.ermaya@serambimekkah.ac.id

Abstract. The main purpose of this study is to identify the chemical components contained in patchouli oil. The method used is water vapor distillation, water distillation. Patchouli plant material used which has been dried by aeration process and chopped above 20 kg. Then it is distilled using several distillation methods. The distillation process is carried out to extract the chemical components of patchouli oil contained in the ingredients. The three distillation methods are used using water because it does not react with patchouli, and can remove patchouli oil chemical components from its patchouli plant tissue, besides that water is also easy to use, has simple set of tools, and has low operating costs. The distillation process is carried out after the material that need to be distilled is put into the kettle and added water then connected to the condenser and heated, then the oil will be condensed and separated with a separating funnel. Analysis using GC-MS was carried out to identify the chemical components of patchouli oil, the result shows that there are 16 constituent components of patchouli oil, with 3 dominant peaks and other components as follow: Patchouli alcohol (42.75%), Delta-Guaiene (28.30%), Azulene (20.48%), Trans Caryophellene (11.84%), Seychellence (CAS) (10.77%), Nephtalene (8.02%), Cycloheptane (6.02%) and Caryophyllene (5, 73%). The main component of patchouli oil is Patchouli alcohol about 42.75% that is obtained from the distillation process using water distillation method with distillation time about 5 hours.

1. Introduction

Patchouli plant (Pogostemon cablin Benth) is one of the essential plant that contribute to foreign exchange market. 90% of patchouli oil around the world is supplied from Indonesia [1]. The development of patchouli oil in Indonesia, especially patchouli production in Nanggrooe Aceh Darussalam (NAD) had contributed about 70% of Indonesian patchouli oil supply or 1696.8 tons / year from an area of 2,876 ha [2]. Indonesian essential oil processing industry has existed since the colonial era. However, evaluated by the quality and quantity it seems there is no significant change at all. This is because most of the essential oil processing units still use simple / traditional technology and generally have limited production capacity.

Patchouli oil is an export commodity that have market opportunity and its existence is needed by various industrial fields both domestic and foreign country [3]. Almost all of Indonesian patchouli oil production is exported especially to the United States, Western European countries, and Japan. The main
component that determines the quality of patchouli oil is patchouli alcohol. Patchouli oil is the main ingredient for binding perfuming agents to the perfume and cosmetics industries. In addition, patchouli oil can be used to control pests [4]. Patchouli oil is widely used in the perfume industry or as an aromatherapy ingredient, because until now there has been no substitute product [5].

Patchouli oil which has high Patchouli alcohol contents can be obtained from the whole patchouli leaves which are chopped first. Patchouli oil in Indonesia is traditionally produced through the process of distillation / distillation of patchouli stems, twigs and leaves. The most dominant component and the active ingredient in patchouli leaves is patchouli alcohol (70-90%) [6].

Generally, the essential oil content in the roots, stems and branches of patchouli plants is smaller (0.4-0.5%), compared to the essential oil content in leaves (2.5-0.5%). _Pogostemon cablin_ Benth, also known as Patchouli Aceh, is widely cultivated in Aceh. Patchouli does not have flowers and leaves are fluffy. Its oil content is (2.5-5.0%). This Acehnese patchouli is one of the highest quality patchouli compared to other varieties.

Patchouli oil distillation conducted conventionally by patchouli farmers that produces patchouli oil with a low yield. Patchouli alcohol content is low and sometimes does not comply the quality standards for patchouli oil as required in SNI (06-2385-2006) so that the price drops. Common obstacles in patchouli agribusiness include low oil content, low oil quality and oil uniformity, as well as the non-continuous products supply and the fluctuating of its prices.

The processing of essential oils from patchouli plant is carried out at Drien Glumbang, Jeumpa and Guhang areas, Kula Batee District, Southwest Aceh. The villages are located at an altitude of 500 meters above sea level with latosol type soil. The processing of essential oils is still very simple, using the steam and water method, the water method and the steam method. This is maybe affects to the oil component produced. Patchouli oil isolation with patchouli alcohol concentration was done by Hernani and Wijaya [7] by extraction process using organic solvents, with a purity of 82.1%. Then, Yanyan et al [8] has carried out the isolation of patchouli oil with patchouli alcohol concentration through three steps, such the fractionation distillation method with low pressure, followed by 1 M NaOH solution, then vacuum liquid chromatography method. The stages of the method can increase patchouli alcohol concentration about 35.77% [9]. To determine the purity of the patchouli oil produced, the best patchouli alcohol concentration was determined through this research.

2. Material and Methods

2.1. Sample Preparations of Patchouli Plant
Patchouli raw material is dried first, drying is need to be done to reduce the moisture content in patchouli plants. After the raw material is dried, cut it into 5 cm pieces using a machete and knife to make it easier for the distillation process of patchouli oil.

2.2. Determination of Water Content
Patchouli raw material is dried on plastic sheeting under the sunlight (30-40°C) protected by trees (not in direct contact with sunlight), then the water content is determined. First the empty cup is dried in an oven at 105°C. Cooled in a desiccator and weighed again. The work was repeated until a constant weight was obtained, then the patchouli plant was weighed about 5 grams in the cup, then dried in oven at 105°C for 3 hours. After that it is cooled in desiccator and weighed again. The work is repeated until a constant weight is obtained.

2.3. Patchouli oil refining process
Ingredients about 20 kg of patchouli leaves, stems and dry twigs (per unit of experiment) are put into a distillation kettle, then heated at temperature 100°C with the appropriate time of treatment. Distillation is
carried out continuously with a pressure less than 1 atm, and distillation time 3, 4, and 5 hours until the distillate is obtained. Refined oil (oil and water) is stored in a container (a closed plastic container). Furthermore, the oil and water stored are separated manually by using a separating funnel. Furthermore, the yield of patchouli oil is calculated and the patchouli oil content is analyzed.

2.4. Gas Chromatography Analysis - Mass Spectrophotometer (GC-MS)
Patchouli oil sample about 1 ml. Injected on a gas chromatography-mass spectrophotometer (Shimadzu GC-MS-QP2010 type) with the operating conditions as follows: the type of column used is Hp-5MS, the column temperature is set at 80ºC for 30 minutes and the speed of temperature increases about 10ºC / minute up to temperature 200ºC and left for 25 minutes. The injector temperature is the same as the detector temperature, which is 310ºC. The carrier gas used is Helium. While the type of electron ionizations is electron impact (EI).

3. Result and discussion
3.1. Quality Standard Analysis Results of Refined Patchouli Oil
Patchouli oil identification analysis was carried out according to SNI 06-2385-2006 [10] about patchouli oil standards. Analysis carried out was patchouli oil concentrations. In accordance with Indonesian quality standards in 2006 there were several parameters used to determine the quality of patchouli oil. These parameters are color, specific gravity, refractive index, solubility in alcohol, and patchouli alcohol content. Patchouli alcohol concentration that identify good or bad quality oils because PA is an active component.

3.2. Gas Chromatography-Mass Spectrophotometer (CG-MS) analysis results
The concentration of patchouli alcohol from GC-MS analysis can be seen in figure 1. The results of the analysis shows the best patchouli oil content from water distillation.

From this data, 16 types of compounds were detected as shown in Table 1 below. Based on Figure 1, there are 16 components identified with 4 highest components which are the components of patchouli oil. From its components of patchouli oil, patchouli alcohol is the most important component in patchouli oil. Because patchouli alcohol has properties that can bind fragrance and then is used in the perfume industry and it has high selling value.

![Chromatogram](image_url)
Table 1. GC-MS data of patchouli oil from water distillation

| Peak Code | Retention Time (tR) | Value (%) | Component Name       |
|-----------|---------------------|-----------|----------------------|
| 7         | 8.765               | 8.02%     | Naphthalene          |
| 11        | 9.898               | 28.30%    | Delta-Guaiene        |
| 8         | 8.995               | 1.44%     | Cis-alpha            |
| 1         | 5.637               | 4.94%     | Methanoazule         |
| 11        | 21.341              | 42.75%    | Patchouli alcohol    |
| 2         | 9.889               | 20.48%    | Azulene              |
| 2         | 7.985               | 11.84%    | Trans-Caryophellene  |
| 6         | 9.577               | 2.31%     | Germacrene           |
| 14        | 24.565              | 6.02%     | Cyclo                |
| 6         | 9.710               | 3.87%     | Bicyclo              |
| 4         | 8.820               | 10.77%    | Seychellen           |
| 3         | 8.714               | 3.53%     | Alpha Patchoulene    |
| 9         | 10.677              | 8.76%     | Delta-Guaiene        |
| 6         | 9.459               | 2.61%     | Caryophyleine        |
| 7         | 9.464               | 3.25%     | Bicyclo [5.3.0]      |
| 6         | 9.604               | 5.73%     | Caryophyllene $B$    |

Table 1 presents the identification of patchouli oil has 16 constituent components of essential oils, with 4 dominant peaks which are compounds: Patchouli alcohol (42.75%), Delta-Guaiene (28.30%), Azulene (20.48%), Trans-Caryophellene (11.84%), Seychellence (CAS) (10.77%), Nephtalene (8.02%), Cycloheptane (6.02%) and Caryophyllene (5.73%).

4. Conclusion
From the results of the study it can be concluded that the results of identification of patchouli oil has 16 constituent components of essential oils, with 4 dominant peaks which are compounds: Patchouli alcohol (42.75%), Delta-Guaiene (28.30%), Azulene (20.48%), Trans-Caryophellene (11.84%), Seychellence (CAS) (10.77%), Nephtalene (8.02%), Cycloheptane (6.02%) and Caryophyllene (5.73%). The main component of the essential oil constituent is Patchouli alcohol compound which is obtained 42.75%.

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