Purification and analysis of patchouli alcohol from patchouli oil by vacuum fractionation distillation

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Abstract: Efforts to purify patchouli alcohol from patchouli oil (Pogostemon cablin Benth.) have been carried out. The patchouli oil was obtained from Bone-Bone village, Baraka sub-district, Enrekang Regency, South Sulawesi, Indonesia. The purification was conducted using vacuum fractionation distillation method with a pressure of 96 kPa. Identification of fractions was carried out by the GCMS method. By this method, four fractions and one raffinate were obtained, namely fraction I (141-142°C), fraction II (145-148°C), fraction III (149-150°C), fraction IV (151-152°C), and raffinate. Patchouli alcohol content in each fraction was 9.76%, 10.86%, 45.37% and 70.34%; while in raffinate was 0%. Based on these results, this method is superior to previous methods, and the fraction IV is suitable to be used as a precursor in derivatization of patchouli alcohol.

1. Introduction

Before being used as a cultivated plant, patchouli (Pogostemon cablin Benth.) is a growing wild plant in Indonesia, Malaysia, China, and India. This herb is used as a perennial herbal concoction that is used to cure various human health problems [1]. This plant contains an oil known as patchouli oil. Patchouli oil was extracted from dried leaves using the steam distillation method [2] and supercritical CO₂ [3].

Indonesia is one of the largest patchouli oil producers in the world. Production of patchouli alcohol estimated about 1200-1300 metric tons per annual, which is more than 90% of the total world production [4]. Based on these figures, it can be said that patchouli oil has a more excellent market prospect compared to other volatile oils.

Patchouli oil is an essential ingredient for the fragrance and cosmetics industry due to the following its properties: (a) volatile than other essential oils, (b) have a bitter taste, (c) soluble in organic solvents such as alcohol, and (d) can be mixed with other essential oils. Because of these properties, then patchouli oils can be used as a fixative for the fragrance industry [5] and as an ingredient in many foods and beverages in the flavoring industry [6].

Patchouli oil containing so many volatile compounds. Patchouli oil from Indonesia contains four major compounds such as seychellene (5.3%), β-patchoulene (5.5%), δ-guaiene (16.7%) and patchouli alcohol (32.2%) [7]. Based on this data, it is known that the most compound content in patchouli oil is patchouli alcohol.

In due to patchouli alcohol has attractive characteristics resembling the aroma of wood and camphor odor and is durable [8], then this compound is used as the main component in the perfume industry and as an essential aroma [9]. Besides, patchouli alcohol is also used in medicine because it can act as an anti-inflammatory [10], anti-influenza [11], and anti-tumorigenic [12].
Based on its chemical structure, patchouli alcohol is a tricyclic sesquiterpene (Figure 1). So many methods have been done to obtain patchouli alcohol with the highest purity, including vacuum redistillation [13], supercritical extraction [14], molecular distillation [15], and microwave air-hydrodistillation [16] however patchouli alcohol purity obtained by these methods is below 40%. These methods also have some disadvantages, including the need for large amounts of energy, using chemicals as solvents so that the results sometimes still containing solvent residues, low yields, and production is still on a small scale.

![Figure 1. Chemical structure of patchouli alcohol](image)

Based on the discussion above, the patchouli alcohol purification in this study was carried out by a different method, namely the vacuum fractionation distillation method. By this method, the patchouli alcohol content in obtained patchouli oil is much higher than that obtained by the previous method.

2. Experimental

2.1 Material
Patchouli oil was obtained directly from patchouli oil farmers in Bone-bone village, Enrekang Regency, South Sulawesi.

2.2 Equipment
Ultra Shimadzu QP2010 Gas Chromatograph Mass Spectrometer coupled with AOC-20i Autosampler using SH-Rxi-5Sil capillary column MS 30 m column length with 0.25 mm inner diameter, vacuum fractionation distillation using a Vigreux column with 20 cm long and 2.4 cm inner.

2.3 Procedure

2.3.1 Vacuum fractionation distillation method. 5 g patchouli oil was inserted into a 250 mL round bottom flask connected to a fractionation column and vacuum pump. The flask was heated over a bath jacket at a temperature of 200°C and a pressure of 96 kPa. Distillates obtained at each change in steam temperature during the distillation process were carried out in 50 mL round bottom flasks, then patchouli alcohol levels of each fraction were measured by the GCMS method.

2.3.2 Determination of patchouli alcohol content using GCMS. The device was regulated under conditions: helium carrier gas, injector temperature 250°C with splitless mode, pressure 76.9 kPa, and carrier gas flow rate 14 mL/min and ratio 1:10. Ion source temperature and interfaces are 200°C and 280°C. Solvent cut time 3 min, range of mass spectrum 400-700 m/z. The initial temperature of the column is 110°C with a hold time of 2 min, and the temperature was increased to 200°C at a rate of 10°C / min and the final temperature is 280°C with a holding time of 9 minutes at a rate of 5°C / min so that the total analysis time was 36 min. Determination of compounds uses the NIST and Wiley libraries.

3. Result and discussion
3.1 Vacuum fractionation distillation

The method of purification with vacuum fractionation distillation gave the best results compared to the previous methods [12-15]. This distillation gave four fractions and a raffinate, as seen in Table 1. In Table 1, it can be seen that the fraction with the most significant yield is fraction II, while the other fractions are almost the same.

Table 1. Fractions of patchouli oil

| Fractions | Temp. (°C) | Weight (g) |
|-----------|-----------|------------|
| I         | 141-142   | 5.57       |
| II        | 145-148   | 26.68      |
| III       | 149-150   | 5.16       |
| IV        | 151-152   | 5.09       |
| Raffinate |           | 2.23       |

3.2 GCMS analysis

Determining the concentration of patchouli alcohol in each obtained fraction were analyzed by the chromatographic method using the GCMS method. Chromatogram of raw patchouli oil, fraction I, fraction II, fraction III, fraction IV, respectively shown in Figure 2, Figure 3, Figure 4, and Figure 5.
In the all obtained chromatograms, each of chromatogram has ten peaks; and based on the NIST and Wiley libraries, each peak is characteristic for compounds as shown in Table 2.
Table 2. Retention times of compounds in all of the fractions

| No. of peak | Retention time (min) | Name of compounds                                      |
|-------------|----------------------|--------------------------------------------------------|
| 1           | 11.203               | β-patchoulene                                           |
| 2           | 12.144               | Seychellene                                             |
| 3           | 12.309               | α-patchoulene                                           |
| 4           | 12.770               | α-bulnensene                                            |
| 5           | 12.840               | Patchoulene                                             |
| 6           | 13.785               | 2,3,3-Trimethyl-2-(3-methyl-buta-1,3-dienyl)-cyclohexanone |
| 7           | 14.406               | Globulol                                                |
| 8           | 14.555               | Epiglobulol                                             |
| 9           | 14.821               | β-selinene                                              |
| 10          | 15.121               | Patchouli alcohol                                       |

The percentage of patchouli alcohol in each fraction is shown in Table 3. In Table 3, it appears that there is an increase in patchouli alcohol content from fraction I to fraction IV. This case, due to patchouli oil contains various components with different boiling points; one of the components with the highest boiling point is patchouli alcohol. In the distillation process, compounds with a low boiling point will evaporate first and then followed by patchouli alcohol, which has a high normal boiling point (287-289°C). In quantitatively, fraction II was the largest fraction compared to the other fractions (Table 1). However, the concentration of patchouli alcohol in this fraction is relatively smaller than fraction IV (Table 3). Therefore, fraction IV is most suitable as a precursor in derivatization patchouli alcohol.

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Table 3. The patchouli alcohol concentration in fractions of patchouli oil

| Fractions | Patchouli Alcohol (%) |
|-----------|-----------------------|
| Raw patchouli oil | 25.30 |
| I         | 9.76                  |
| II        | 10.86                 |
| III       | 45.37                 |
| IV        | 70.34                 |

4. Conclusion
Patchouli alcohol can be purified from patchouli oil using a vacuum fractionation distillation method. This method increases the purity of patchouli oil from 25.30% to 70.34%. This method is more effective in the separation and purification of patchouli alcohol than the previous methods. This method gave fraction IV, which suitable to be used as a precursor in derivatization patchouli alcohol.

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