The fractional separation of citronella, cajeput, and patchouli crude oils using spinning band distillation

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Abstract. Spinning band distillation is one of the methods to separate high and close boiling mixtures. Spinning band distillation can be effective as it does not use solvent or other additive for the separation. Fractionation of Citronella, Cajeput, Patchouli essential oils has been experimentally conducted using B/R Instrument Spinning Band Distillation System Model 36-100. The crude oil was placed in the 150 mL heating pot and the temperature cut and heating rate was set. The range of temperature cut is around 230-300, 176-280, and 150-230°C for Patchouli, Citronella, and Cajeput, respectively. Running pressure was set at 10 mmHg for Patcouli oil, and 30 mmHg for citronella and cajeput oil. As expected, the heating process was stable and the solution manages to boil. It is observed that using spinning band distillation, the condensed vapor flow faster than simple vacuum fractionation and at the end product was maximize. The citronella, 1,8-cineole, and patchouli alcohol component were 88%, 67%, 44%, respectively.

1. Introduction
A large number of plants have been performing well for extraordinary bioactivities [1-4]. This benefit is important to acknowledge those plants for medicinal uses [4-6]. The activities of essential oil, a volatile component, provide more secure application. Various methods for isolating essential oil component from plants have been explored [7-11]. The use of organic solvent and steam distillation are two of the most uses method for extracting the essential oils. In advanced, the crude extract should be purified to improve the bioactivities as well as an economical value.

Separation process of essential oil can be carried out using fractional distillation. The problem of fractional distillation is the close boiling value of each components [12, 13]. However, this issue can be overcome by using high theoretical plates of fractional column. Spinning band distillation is a distillation technique combined with rotating helix band to generate high theoretical plates. The high number of theoretical plates during distillation is ideal to get very good separation of mixed components [14]. The separation of essential oils, crude oil and solvent recycling are oftenly use spinning band distillation to get the high purity fractions. Spinning band column usually made from metal or Teflon. The former spinning band is good for preparing samples that relatively have broad and high boiling points. The second one, perform well for distillation below 225°C. The band is tightly fitted to amarture bore of glass tube and rotated at a fast pace.

The most important Indonesian herbal and aromatic plants containing essential oil for high-end commercial production and marginally exploited are cloves (Syzygium aromaticum), cajuput (Melaleuca leucadendron), lemongrass (Cympobogon citratus), turpentin (Pinus pinacea) and patchouli
(Pogestemon cablin). Our previous studies were also related to those plants. The purification were conducted using conventional vacuum fractional distillation. The highest yield obtained from this method was 80.58% [15], 34.96% [16], 75.67% [17] and 88.79% [16], for cloves oil, cajuput oil, lemongrass oil, and patchouli oil, respectively. In order to improve the quality and shorten the distillation time, current study focussed on the use of spin band distillation for separating essential oil components from crude Citronella, Cajeput, Patchouli oils.

2. Materials and Methods

2.1. Materials

The crude oil of Citronella, Cajeput, Patchouli was used as a raw material. The crude oil was commercially available and obtained from different company in Jawa island. Acetone and hexane were used as a cleaning agent of glassware.

Separation of crude oils was carried out with B/R Instrument-Spinning Band Distillation System Model 36-100 integrated with programmable controller BR M690. GC-MS QP2010S from Shimadzu was used to characterize the component of each samples.

2.2. Methods

A 150 mL of crude oil was poured into boiling flask and connected to the fractional column (120 cm). The fractionating tower was operated in vacuum using a PC BRM690. The distillation set was shown in Figure 1 as well as the controlling parameter shown in Table 1.

Figure 1. B/R Instrument Spinning Band Distillation System Model 36-100.
Table 1. Set up parameter of fractional distillation using spinning band distillation.

| Instrumental set up | Citronella | Patchouli | Cajeput |
|---------------------|------------|-----------|---------|
| Run Pressure (mmHg) | 30         | 30        | 10      |
| Initial heat (%)    | 20         | 20        | 20      |
| Equilibration (minutes) | 10      | 10        | 15      |
| Number of cut       | 3          | 5         | 7       |
| Max Pot Temp (°C)   | 300        | 300       | 230     |
| Condenser Temp (°C) | 30         | 30        | 35      |
| Real Run Pressure (mmHg) | 29.9-30.1 | 29.9-30.1 | 9.64-10.7 |

3. Result and Discussion

Essential oils are volatile oils distilled from plant materials and represent the typical flavour and aroma of a particular plants. They can be found in bark, leaves, flowers, and seeds. The essential oil content of plant material is low, usually it only containing 1-3% of the plant weight. Moreover, their low-volume make them very high value products. Correct harvesting method is then very important. The essential oil content varies considerably during the development of the plant and even the time of day. If the plant is harvested at the wrong time, the oil yield or its quality can be severely reduced.

Secondly, the value of essential oil will improve if the quality (purity) of the oil is high. Distillation is a method of separating liquids in a mixture of solution by based on their boiling points. It has many commercial applications. There are specific types of distillation based on the specific mechanism used in the process.

3.1. Citronella oil fraction

The crude citronella oil appearance was yellowish liquid with viscosity of 4.1 Cst (at 25 °C). According to the data of GC-MS analysis, it is found that the lemongrass crude oil containing mostly terpenoid compounds. The main component of Java citronella oil is citronellal of 43%. Further, the Spinning band fractional distillation provide better yield up to 88% (4th fraction) as it shown in Figure 2.

Figure 2. (a) Chromatogram of 4th fraction of Citronella oil and (b) distillate fractions of cajeput oil.
3.2. Cajeput oil fraction
The crude Cajuput oil appearance was green to yellow liquid with viscosity of 2.2 Cst (at 25 °C). The chromatogram of crude oil indicate the content of 1.8 sinalol of 51%. In advanced, after fractional distillation using spinning band distillation instrument, the yield of 1.8 sinalol was improved up to 67% (1st fraction) as it shown in Figure 3.

![Figure 3](image)

**Figure 3.** (a) Chromatogram of 1st fraction of cajeput oil and (b) distillate fractions of cajeput oil.

3.3. Patchouli oil fraction
The crude Cajuput oil appearance was dark yellow liquid with viscosity of 4.9 Cst (at 25 oC). The chromatogram of crude oil indicate the content of patchouli alcohol of 41%. further, the spinning band fractional distillation yield high content of patchouli up to 64% (4th fraction) as it shown in Figure 4.

![Figure 4](image)

**Figure 4 (a)** Chromatogram of 1st fraction of patchouli oil and (b) distillate fractions of patchouli oil.
Fractional distillation is the separation of a mixture into its fractions, or component parts. As the solution to be purified is heated, its vapors rise to the fractionating column. As it rises, it cools, condensing on the condenser walls and the surfaces of the packing material. In this study, we found that the setup of spinning band distillation instrument is optimized for citronella oil. On the other hand, cajeput oil and patchouli oil provide low level purity of 1,8-cineole and patchouli alcohol content, respectively. The boiling cycle at a given temperature does not occur at exactly the same position in the fractionating column; theoretical plate is thus a concept rather than an accurate description. A spinning band distillation system uses a spinning band of Teflon or metal to force the rising vapors into close contact with the descending condensate, increasing the number of theoretical plates.

4. Conclusion
Spinband fractional distillation was successfully used to separate components from citronella, simeo, and patchouli crude oils. The controlled setup at the instrument affect significantly to the performance of fractional separation. Citronellal was obtained at high-level of yield (88%). However, the set up parameter for fractional distillation of cajeput and patchouli alcohol is still need to be optimized to get high yield purity.

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