Physico-Chemical Properties of Patchouli Oils (*Pogostemon cablin*) Separated by Fractional Distillation Method

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Abstract. Patchouli (*Pogostemon cablin)* is one species of the vegetation that produces essential oil known as patchouli oil. The quantity of patchouli alcohol contained in patchouli oil determines the oil quality. This study investigates fractional distillation of patchouli oils (*Pogostemon cablin*) to increase the level of patchouli alcohol in patchouli oils. The separation process is carried out by fractional distillation method applying four fractionation temperatures: 120°C, 125°C, 130°C, and 135°C. The compositions of each fraction were identified using Gas Chromatography – Mass Spectrometry (GC-MS). Patchouli oil A and B with initial patchouli alcohol content of 27.03% and 36.87% were successfully fractionated resulting four fractions of patchouli oils. The final levels of patchouli alcohol are increased to become 35.35% and 43.62%, respectively as well as the densities of the four fractions.
Keywords : Patchouli oil, fractionation, patchouli alcohol

Introduction

Patchouli oil is an essential oil extracted from patchouli (*Pogostemon cablin*) leaves by distillation or other methods such as solvent extraction method and supercritical CO_2 (Donelian *et al*, 2009) . Patchouli oil is one of Indonesian most important commodity for export purpose. This essential oil is always in high demand to be traded internationally and added as an essential ingredient for perfumery industries, cosmetics, soaps, detergents, and pharmaceuticals.

A wide range of industrial application of patchouli oil is particularly due to its typical strong scent and most importantly its function as a fixative agent. A fixative agent is a substance used to reduce the rate of evaporation and is able to increase the mixture stability when added to more volatile components. The fixative properties of patchouli oil are mainly contributed by patchouli alcohol ($C_{15}H_{26}O$) as the main component which can classified into oxygenated terpenes (Dummond, 1960). Other major constituents in patchouli oil are δ-guaiene, α-guaiena, seychellene, α-patchoulene, carryphylene, β-patchoulene, pogostol, and norpatchoulenol (Akhila *et al*, 1987).

Typical patchouli oil extracted by farmers has low level of patchouli alcohol commonly below 30%. This low grade oil consequently produces patchouli oil with low market price. The level of major components in patchouli oil can be increased by appropriate determination of fractional distillation temperatures and pressures.

The aims of this research were to investigate patchouli oils separation by fractional distillation method. In particular, the effect of increasing distillation temperatures toward the oils' composition and their physico-chemical properties were also evaluated.

Materials and Methods

Materials

The patchouli oil was purchased from local farmer in Tapaktuan, South of Aceh District, Aceh Province, Indonesia. As a common practice, the patchouli oil was extracted from patchouli leaves by steam distillation.

The fractional distillation equipments consist of: heated oil bath, round bottom distillation flask, fractionating column, condenser, conical receiving flask, distillation adaptors, and thermometer.

Methods

Fractionation of patchouli oil procedure

Patchouli oils from two sources (patchouli oil A and B) were used in which each of them has different initial quantity of patchouli alcohol (PA): PA-1, 27.03% and PA-2, 36.87%. Separation of patchouli oils into their fraction were carried out by fractional distillation method. Two variables were examined, initial content of patchouli alcohol (27.03%; 36.87%) and fractionation temperatures (120°C, 125°C, 130°C, 135°C). Different initial values might influence the final patchouli alcohol fraction. Fractional distillations were performed at 100 mbar.

The process was started by filling 250 g patchouli oil into round-bottom distillation flask. The temperature was set according to the mentioned variables. The cooking oil was used as the medium for heat transfer. Once the boiling temperature has reached, certain amount of fraction was collected in receiving flask. The whole process separated distillate oils from their residues. The residues' physico-chemical properties were determined following the parameters set by the "Standar Nasional Indonesia" (SNI) while the chemical compositions were identified using GC-MS.

Measurement of oil physico-chemical properties

Measurement of the oil physico-chemical properties including specific weight, refractive index, optical rotation, solubility in alcohol, acid value, ester value, and determination of patchouli alcohol content. All of these parameters are described in the SNI 06-2385-2006 (Badan Standarisasi Nasional, 2006).

Identification of chemical composition and chromatographic analysis

Identification of chemical composition is determined by gas chromatography–mass spectrometry (GC-MS) instrument. The GC analysis of the oils was performed on gas chromatograph, fitted with a Stabilwax capillary column, 30 m x 0.25 mm x 0.25 um, and a flame ionization detector (FID). Helium was the carrier gas, performed under split injection mode. The oven temperature was set initially at 100 °C for 2 min and increase to 160 °C at the rate of 5°C/min for 5 min, then to 220 °C at the rate of 10 °C/min and hold for another 10 min.

The GC-MS analysis was performed on a gas chromatograph (Shimadzu GCMS-QP 20105), using a fused-silica capillary column (Rtx-5MS), coupled to a selective mass detector. The injector temperature was 300°C. The initial oven temperature was 80°C. The carrier gas was helium with a flow rate of 0.68 mL/min. The sample volume injected was 1 µl with a split rate of 99.7. Identification of each fraction of the patchouli oil was based on the comparison with GC-MS library (Electronic Wiley Library).

Results and Discussion

The Physico-Chemical Properties of Patchouli Oil

Table 1 shows the results of the physico-chemical properties measurements of patchouli oil A and B including specific weight, refractive index, optical rotation, solubility in alcohol, acid value, esther value, patchouli alcohol content and alpha copaene content. All the values obtained are in the range and met the quality standard approved by the SNI.

The results of GC-MS analysis of initial patchouli oil A and B are written in Table 2. The chemical composition reveals that the patchouli oil used in this experiment is consist of 15 main components. Five components with the highest percentages are patchouli alcohol (27.03 % and 36.87%), δ-guaiene (16.35% and 15.02%), α-guaiene (14.15% and 10.41%), seychellene (8.25% and 6.51%), and α-patchoulene (5.94% and 4.92%). This finding is in agreement with what have been found by Corine and Selliers (2004). They also described five major components of the patchouli oil as patchouli alcohol (32.2%), δ-guaiene (16.7%), α-guaiene (15.6%), seychellene (5.3%), and α-patchoulene (5.5%).

Table 1. The physico-chemical properties of patchouli oil A and B

Parameters	Patchouli oil A	Patchouli oil B
Colour	yellow-brown	yellow-brown
Specific weight (25°C/ 25°C)	0.964	0.965
Refractive index ($_nD^{20}$)	1.513	1.515
Solubility in alcohol 90%	Clear solution in volume ratio of 1:10	Clear solution in volume ratio of 1:10
Acid value	0.50	0.52
Esther value	12.62	12.65
Optical rotation	(-) 52°	(-) 53°
Patchouli Alcohol	27.03%	36.87%
Alpha copaene (%)	Not detected	Not detected

Table 2. Composition of patchouli oil A and B

Peak Number	Retention Time (min)	Compound	Patchouli oil A (%)	Patchouli oil B (%)
1	10.19	β-patchoulene	3.32	2.51
2	10.34	β –elemene	1.25	0.93
3	10.89	Seychellene	0.93	0.54
4	11.09	trans-caryophyllene	4.61	2.96
5	11.54	α-guaiene	14.15	10.41
6	15.26	seychellene	8.25	6.51
7	11.92	α-humulene	1.07	0.73
8	12.04	α-patchoulene	5.94	4.92
9	12.11	δ-gurjunene	2.69	2.14
10	12.17	Patchoulene	1.13	0.93
11	12.26	trans-caryophyllene	0.70	0.54
12	12.42	α-guaiene	0.81	0.65
13	12.74	Eremophilene	0.95	0.79
14	12.97	α-guaiene	5.35	0.63
15	13.20	δ-guaiene	16.35	15.02
16	15.10	caryphyllene	0.74	0.96
17	16.26	benzocyclohepten	1.08	1.23
18	17.31	veridiflorol	2.78	4.51
19	17.63	patchouli alcohol	27.03	36.87
20	19.47	2H-pyran-2-one	0.87	1.02

Fractional Distillation of Patchouli Oil

The four fractions resulted from fractional distillation of patchouli oils were found to be correlated with the patchouli alcohol content. The first three fractions (fraction 1, 2, and 3) showed lower values as indicated in Table 3. The highest patchouli alcohol contents were collected from the 4th fractions, 35.45% and 43.59% for patchouli oil A and patchouli oil B, respectively. These outcomes were the consequences of other components of patchouli oil that have been vaporized in the previous fractions. Therefore, higher amount of patchouli oil is considered due to the reduction of other components.

Table 3. Results of fractional distillation of patchouli oil A

Patchouli Oil Components and Its fractions (%)						
Retention time	Main Components	Initial Oil	F1	F2	F2	F4
17.63	Patchouli alcohol	27.03	17.46	28.21	29.23	35.45
13.20	δ-guaiene	16.35	9.33	16.33	17.32	13.91
11.54	α-guaiene	14.15	16,19	13.40	13.86	9.80
11.74	Seychellene	8.25	19.36	7.95	8.04	6.39
12.04	α-patchoulene	5.94	9.81	5.95	5.99	4.85

Table 3 are evidence for increasing percentage of patchouli alcohol separated from patchouli oil A: fraction 1 (17.46%), fraction 2 (28.21%), fraction 3 (29.23%), and fraction 4 (35.45%). Conversely, the level of δ-guaiene, α-guaiene, seychellene, and α-patchoulene were decreased as the fractional distillation temperatures increased from 120°C, 125°C, 130°C, and to become 135°C.

Table 4. Results of fractional distillation of patchouli oil B

Patchouli Oil Components and Its fractions (%)						
Retention time	Main Components	Initial Oil	F1	F2	F2	F4
17.69	Patchouli alkohol	36.87	9,88	27.03	36.79	43.62
13.19	δ-guaiene	20.07	28.3	26.4	24.1	15.8
11.53	α-guaiene	15.91	23.1	18.2	10.4	7.5
11.73	seychellene	6.95	12.3	9.8	6.3	4.8
12.03	α-patchoulene	2.34	7.8	6.4	3.8	1.4

Extraction analysis of patchouli oil B demonstrated similar trend as can be seen in Table 4 where patchouli alcohol contents were inclined from fraction 1 to fraction 4. The remaining components were declining accordingly: δ-guaiene (from 20.07% to 15.80%), α-guaiene (from 15.91% to 7.5%), seychellene (from 6.95% to 4.8%), and α-patchoulene (from 2.34 to 1.4%).

The findings imply that lower temperature (120°C) produced new distillate (fraction 1) containing more components which can be categorized as lighter substances such as seychellene and α-patchoulene. These two substances are easily to vaporize than others. In addition, the middle group components (δ-guaiene dan α-guaiene) are also found significantly. Increasing temperatures from 120°C to: 125°C (fraction 2), 130°C (fraction 3), and to become 135°C (fraction 4) exhibit the same patterns. Patchouli alcohol contents as the main component are increasing whereas the middle and lighter substances are decreasing. Above explanation is also supported by the results of patchouli oil physico-chemical properties as illustrated in Table 5 and 6 of Patchouli oil A and B, particularly the oil density (specific weight).

Table 5. The physico-chemical properties of patchouli oil A

Parameters	Initial Oil	F1	F2	F3	F4
Density (Specific weight)	0.965	0.9610	0.9690	0.9641	0.9681
Optical rotation (α)	(-48.43^o) – (-59.65^o)	(-49.43^o) – (-59.65^o)	(-49.85^o) – (-62.63^o)	(-49.98^o) – (-63.51^o)	(-49.98^o) – (-63.95^o)
Colour	Brown yellow	Light yellow	Light yellow	Light yellow	Light yellow

Table 6. The physico-chemical properties of patchouli oil B

Parameters	Initial Oil	F1	F2	F3	F4
Density (specific weight)	0.964	0.9410	0.9490	0.9641	0.981
Optical rotation (α)	(-48.25) – (-60.13^o)	(-48.43^o) – (-60.47^o)	(-48.85^o) – (-60.63^o)	(-49.18^o) – (-61.32^o)	(-49.26^o) – (-61.53^o)
Colour	Brown yellow	Light yellow	Light yellow	Light yellow	Light yellow

The initial content of patchouli alcohol is found to be correlated with the final content of patchouli alcohol in fraction 4. Higher initial content of patchouli alcohol in the original oil produce elevated level of patchouli alcohol after the 4th fractional distillation step at 135°C. Initial patchouli alcohol of patchouli oil A which was 27.03% could be improved to become 35.45% whereas 36.87% patchouli alcohol of patchouli oil B was purified to become 43.62%.

Conclusions

Results of GC-MS analyzes confirm that patchouli oils consist of five main components, namely: patchouli alcohol, δ-guaiene, α-guaiene, seychellene, and α-patchoulene. Rising fractional distillation temperatures of patchouli oils from 120°C, 125°C, 130°C, and to become 135°C produce increasing quantity of patchouli alcohol in each fraction but decreasing the amount of δ-guaiene, α-guaiene, seychellene, and α-patchoulene. The initial content of patchouli alcohol in the original patchouli oil influences the final quantity of patchouli alcohol.

References

Akhila, A., Sharma, P.K., and Thakur, R.S., 1987. 1,2-Hydrogen shifts during the biosynthesis of patchoulenes in Pogostemon cablin. Phytochemistry, 26, 2705 – 2707.

Corine, M.B., and Sellier, N.M., 2004, Analysis of the essential oil of Indonesian patchouli (Pogostemon cablin Benth.) using GC/MS (EI/CI). J. Essent. Oil Res, 3, 16-17.

Donelian, A., Carlson, L.H.C., Lopes, T.J., and Machado, R.A.F., 2009. Comparison of extraction of patchouli (Pogostemon cablin) essential oil with supercritical CO_2 and by steam distillation. J. Supercritical Fluids, 2, 15-20.

Dummond, H.M., 1960, Patchouli oil, J. Perfumery and Essential Oil, 484-493.

Standar Nasional Indonesia, 2006. Standar Minyak Nilam. No. 06-2385-2006. Jakarta