Proceedings of The 3rd Annual International Conference Syiah Kuala University (AIC Unsyiah) 2013
In conjunction with The 2nd International Conference on Multidisciplinary Research (ICMR) 2013
October 2-4, 2013, Banda Aceh, Indonesia

Increment of patchouli alcohol in patchouli oil by vacuum distillation fraction method

Yuliani Aisyah, Sri Haryani Anwar and Yulia Annisa
Agricultural Product Technology Department, Syiah Kuala University,
Banda Aceh 23111, Indonesia.
Correspondeng Author: yuli_stp@yahoo.com

Abstract. The quality of Indonesian patchouli oil is commonly lower than that required by standard (substandard). The quality of patchouli oil is determined by the level of an organic compound known as patchouli alcohol (PA). This research is aimed to study the influence of the initial level of patchouli alcohol in patchouli oil to the final oil quality after being separated by vacuum distillation fraction method. The patchouli oils used had three different initial level of patchouli alcohol (31.11%, 32.83% and 33.61%). The results show that the increments of the initial level of patchouli alcohol tend to lower the final concentrations of this compound in the patchouli oil. The oil with 31.11% of initial patchouli alcohol contained 49.07% PA after distillation, the initial PA of 32.83% became 42.87% and the oil with the highest initial PA (33.61%) consisted only 32.85% PA in its final quality.
Keywords: patchouli oil, patchouli alcohol, vacuum distillation fraction, essential oil quality

Introduction

Patchouli (*Pogostemon patchouli*; also known as *Pogostemoncablin*Benth.) is herbaceous plants with leafy shrubs and quadrangle trunked. The dried leaves and stalk of this plants can distilled to get patchouli oil (Mangun, 2005). Need for patchouli oil in the world reaches 500-550 tons per year and Indonesia can meet up to 450 tons per year. However, Indonesia's patchouli industry is constrained by the poor quality of the produced oil. Most chemical properties determine the quality of patchouli oil is patchouli alcohol levels (PA). According to Trifilief (1980), patchouli alcohol is the largest component in patchouli oil and plays a role in gives out the distinctive odour in patchouli oil. During this time the farmer was only able to produce patchouli oil with 26-28% patchouli alcohol.

Meanwhile the factory distillation with steel distillation equipmentis capable to produce patchouli oil with 31-35 % of patchouli alcohol. The content of the active compounds can be improved, either by using the method of vacuum distillation fractionation. According to Guenthers (1987), essential oil fractionation is separation of volatile oil into several fraction based on boiling points differences. This process aimed to purify a substance from compounds which having boiling points nearly as to it. If a liquid distilled unstable at certain temperature, or if its boiling point at normal condition is too high, the distillation may be done in vacuum condition (Irawan, 2010). Vacuum is a condition of the air/gas around a particular environment which the air pressure is in its neighborhood are under atmosphere pressure.

In general, this research is intended to improve the quality of patchouli oil by using the vacuum distillation fraction method through the increment of patchouli alcohol. In particular, this research aims to know the influence of initial levels of patchouli alcohol against increment of patchouli alcohol in patchouli oil.

Materials and Methods

Research done in the laboratory of vegetable Processing and forest products Processing Laboratory which located at agricultural faculty iniSyiah Kuala University. Analysis of patchouli alcohol levels was carried out in the laboratory of organic chemistry, Faculty of Mathematics and natural sciences at GadjahMada University. Research underway since March, 2013 until June 2013.

The patchouli oil in this research obtained from South Aceh district, sub-district Meukek, Pasie Raja and panjupian. This research also used cooking oil as heat conductormedium. The equipment used in this study was a series of vacuum fraction

distillation apparatus consisting of a round-neck flask of 500 ml size, Vigreux column (35 cm hight), thermometer, condenser, 3 heart flask, vacuum pumps, pot and hot plate and also the quality analysis tools consisting of Gas Chromatography Mass Spectrometry (GC-MS)Shimadzu GCMS-QP 2010S and Gas Chromatography 2010S GC-QP.

Methods

1. Vacuum Fraction Distillation Process (Modified Aisyah, 2008)

Patchouli oils from three sources (patchouli oil A, B and C) were used in which each of them has different initial quantity of patchouli alcohol (PA): Patchouli oil-1, 33,11%, Patchouli oil-2, 32,83% and Patchouli oil-3, 33.61%. Separation of patchouli oils into their fraction were carried out by fractional distillation method. Different initial values might influence the final patchouli alcohol fraction. Fractional distillations were performed at ±2 KPa (± 15,001 mmHg) and temperature 130-190°C.

The process was started by filling 300 ml patchouli oil into round-bottom distillation flask 500 ml size. Once the boiling temperature has reached, certain amount of fraction was collected in receiving flask. A column fractionate used having a high 35 cm. Distillation was carried out until no more distillate drips into the heart 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.

2. 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).

3. Identification of chemical composition and chromatographic analysis

Identification of chemical composition is determined by gas chromatography–mass spectrometry (GC-MS) and gas chromatography instrument.

a. GC-MS

GC analysis of the oil was performed on Shimadzu GCMS-QP 2010S with a FID detector and helium as a carrier gas with 40 ml/min flow rate. The instrument was fitted with a 30 m x 0.25 mm non-polar AGILENTJ%W DB-1column. Oven temperature was programmed from 70°-260°C at 5°C/min, held at 260°C for 17 minutes. Injector temperature was 310°C. The analysis was done to patchouli oil before fractionated. Identification of each fraction of the patchouli oil was based on the comparison with GC-MS library (Electronic Wiley Library).

b. GC

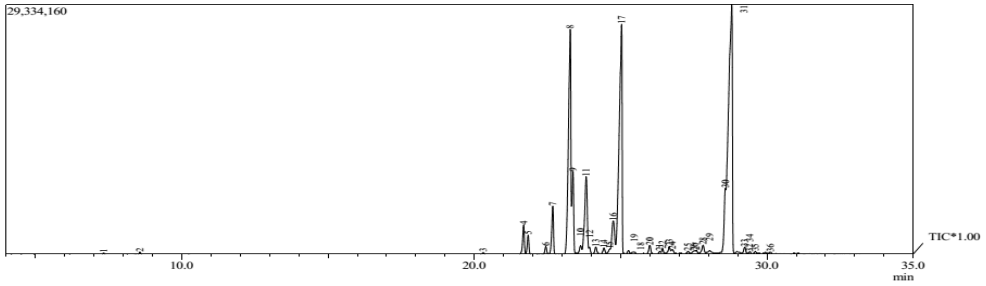
This method was used for identification of the components residue fraction from vacuum fraction distillation. GC analysis was carried out with a Shimadzu GC-QP 2010S GC under the following conditions: as much as 0.2 ml sample was injected into the injector T280, column used was CBP1, helium used as carrier gas with flow rate of 40 ml / min and detector used was FID T300. The temperature conditions used were the same as those given above for the GC-MS analysis.

Results and Discussion

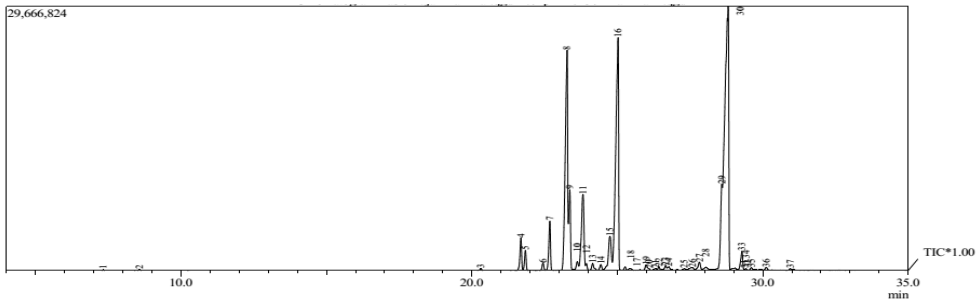
Patchouli Oil Components

Before the vacuum distillation fractionation, patchouli oil which will fractionate first analyzed levels of patchouli alcohol. From the analysis of GC-MS can be seen that all three of patchouli oil has different levels of patchouli alcohol. Besides patchouli alcohol, patchouli oil also dominated (± 85%) by other components such as *Δ-guaiene, α-guaiene, α-patchoulene, seychellene*and *β-carryophyllene.* The patchouli alcohol levels and other components was different in each oil. Such differences may be caused by several factors such as genetic (species), aquaculture, environment, harvest and post-harvest handling (Irawan, 2010).

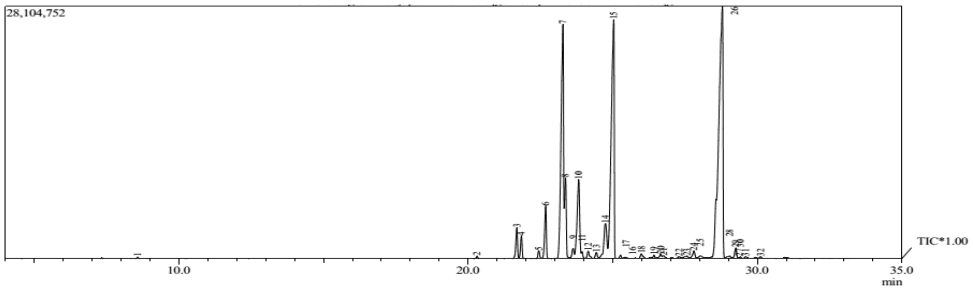
Those components were slightly different from the analysis results of Corine and Sellier (2004). On their analysis found four new components which are *γ-gurjunene, germacrene D, aciphyllene* dan *7-epi-α-selinene*. While the results of the oil analysis in this research only found γ-gurjunene and *germacrene A*. In addition to the factors mentioned earlier, this difference is also thought to be caused by a GC-MS analytical methods used are different. The results of GC-MS analysis of initial patchouli oil A, B and C are written in Figure 1.



(Patchouli oil-1)



(Patchouli oil-2)



(Patchouli oil-3)

Figure 1. Chromatogram of patchouli alcohol with initial patchouli alcohol levels 31.11% (patchouli oil-1), 32.83% (patchouli oil-2), 33.61% (patchouli oil-3).

The peak patchouli alcohol in patchouli oil chromatogram is located at the end of the chromatogram. This shows that patchouli alcohol is a component that has a high boiling

point in patchouli oil compound classes other than terpenes. Relatively high boiling point may explain why patchouli oil has fixative properties. Based on the results chromatogram, patchouli alcohol content of 31.11% at retention time 28.794 minute (patchouli oil-1), 32.83% at retention time 28.810 minute (patchouli oil-2), and 32.83% at retention time 28.810 minute (patchouli oil-3). Peaks that do not split in the GC-MS test conditions shows the components in the patchouli oil relatively stable.

The chemical composition reveals that the patchouli oil used in this experiment is consist of 36 main components (patchouli oil A), 37 main components (patchouli oil B), and 32 main components (patchouli oil C). Five components with the highest percentages are patchouli alcohol (31.11 %), δ-guaiene (21.45 %), α-guaiene (17.39 %), α-patchoulene (6.89 %), and seychellene (4.68 %). This finding is in agreement with what have been found by Dung *et al* (1989). They also desribed five major components of the patchouli oil as patchouli alcohol (37.8%), δ-guaiene (14.7%), α-guaiene (13.4%), α-patchoulene (8.0%), and seychellene (7.5%). Corine and Selliers (2004) 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%).

Patchouli Alcohol Levels

From the results of the analysis revealed that the final levels of patchouli alcohol tends to decrease along with the rising initial levels of patchouli alcohol. The GCMS and GC analysis result of patchouli alcohol levels are listed in Table 1.

Table 1. The GC-MS and GC analysis result of patchouli alcohol levels

Before Fractionated (GCMS)	After Fractionated (GC)
31,11%	49.07%
32,83%	42.87%
33,61%	32.85%

The occurrence allegedly because of patchouli oil that has a low initial levels of patchouli alcohol still contains many low-boiling point components. It can be seen in the analysis report of chemical components using GC-MS (Figure 1, 2 and 3), patchouli oil which had 31.11% patchouli alcohol has 36 peaks which indicates that the patchouli oil contains many types of components. At the time of fractionation distillation process takes place, these components largely evaporate and become distillate. So the high-boiling component such as patchouli alcohol was left in the residue. While the patchouli oil that has high initial patchouli alcohol levels, the number of components low-boiling is slightly (patchouli oil with 33.61% concentration of patchouli alcohol only have 32 peaks) so that the components that can be evaporated was modest. Therefore the increment of patchouli alcohol in this raw patchouli oil was not too high.

Physical Properties Analysis

Before fractionated, the physical properties of raw patchouli oil was analyzed. This analysis took the patchouli oil with 31.11% patchouli alcohol concentration. Table 2. shows the results of the physical properties measurements of patchouli oil A (before and after fractionated) including specific weight, refractive index, and solubility in alcohol. All the values obtained are in the range and met the quality standard approved by the SNI.

Table 2. The physical properties of patchouli oil-1, 2 and 3

Parameters	Before fractionated	After fractionated

Specific weight (25°C/ 25°C)	0.954	0.979
Refractive index ($_nD^{20}$)	1.5070	1.5115
Solubility in alcohol 90%	Clear solution in volume ratio of 1:8	Clear solution in volume ratio of 1:7

The results shows that the oil had 0.953 specific gravity, 1.5070 refractive index and soluble in ethanol 90% at 1:8 ratio. Some analysis was also done to one of residue fraction, ie the residue with 49.07% final concentration of patchouli alcohol. The results shows that the residue fraction had 0.979 specific gravity, 1.5116 refractive index and soluble in ethanol 90% at 1:7 ratio. The report indicates that the specific gravity and refractive index point from residue fraction was increase. This tendency thought to because of the patchouli alcohol increment. As known before the physical properties affected by components of the oil. Patchouli alcohol is a long-chain molecul component, this component also have a functional cluster (Armando, 2009 and Guenther, 1987).

Conclusions

Results of GC-MS analyzes confirm that patchouli oils consist of five main components, namely: patchouli alcohol, δ-guaiene, α-guaiene, α-patchoulene and α-patchoulene. The final levels of patchouli alcohol tends to decrease along with the rising initial levels of patchouli alcohol. The patchouli alcohol concentration also influence the physical properties of the oil.

References

Aisyah, Y., Hastuti, P., Sastrohamidjojo, H., Hidayat, C. 2008. *Komposisi Kimia dan Sifat Anti bakteri Minyak nilam (Pogostemon cablin).*Majalah Farmasi Indonesia, 19 (3), 151-156.

Armando, R. 2009. *Memproduksi 15 Jenis Minyak Atsiri Berkualitas*. Penebar Swadaya, Jakarta.

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.

Dung, N.X., Leclercq, P.A., Thain, T.H., and Moi, L.D., 1989, Chemical composition of patchouli oil from Vietnam. J. Essent. Oil Research, 3, 1-2.

Guenther, E. 1987. *Essential Oils*. Van Nostrand Reinhold Company, New York.

Irawan, B. 2010. *Peningkatan Mutu Minyak Nilam dengan Ekstraksi dan Destilasi Pada Berbagai Komposisi Pelarut.* Tesis. Magister Teknik Kimia Universitas Diponegoro, Semarang.

Mangun, H.M.S. 2005. *Nilam*. Penebar Swadaya, Jakarta.

Trifilieff, E., 1980. *Isolation of the Postulated Precursor of Nor-patchoulenol in Patchouli Leaves*. *J. Phytochemistry*, 19, 331 – 332.