Identification of Patchouli – Chemical Properties on Oil Purification by Using Acid-Activated Bentonite

Netty Sri Indeswari #
# Majoring in Agricultural Products of Technology, Faculty of Agricultural Technology, Andalas University, Padang, Indonesia
E-mail: nettibasnafdi@gmail.com

Abstract—This research is aimed at obtaining oil chemical properties on purification that include solubility in alcohol, total acid, total ester, Fe Content, Patchouli Alcohol Content, Alpha Copae (C15H24) Content, and purity. The method of research used is Complete Randomized Design (CRD) with factorial design. Two factors have been examined are 1) bentonite activations with 3 levels; inactivated bentonite, H2SO4-activated bentonite, and HCl-activated bentonite; and 2) the number of bentonite applications with 5 levels: 1%, 2%, 3%, 4%, and 5%. And analysis of oil color is made by using UV spectrophotometer to see clearness or its transmittance percentage. Acid-activated bentonite by number of bentonite given affects ethanol solubility, total acid, total ester, Fe content, Alpha copae content, and patchouli alcohol content. H2SO4-Activated bentonite by 2% given was indicated as the best result by increasing percentage of transmittance from 69% to 81.550%, ethanol solubility 4,050%, total acid 2,120, total ester 7,975, Alpha copae content 0.04%, Patchouli alcohol content 25.02%, and Fe content 5,864 mg/kg.

Keywords—Bentonite; Patchouli Oil; Purification

I. INTRODUCTION

Patchouli oil is one of popular commodities in oil trading world. It is extracted from distilling the leaves and trees of patchouli plants Pogostemon cablin Benth. This oil is known as a fragrant aroma fixative and prevents evaporation of fragrant gases in perfume.

In 2008, the whole area of patchouli plantation in West Sumatra, Indonesia, was 3,042 Ha concentrated on Pasaman Barat regency with 1,315 Ha in area, followed by Mentawai regency with 990 Ha in area.

In recent years, the patchouli oil produced by local farmers has very low grade, making the selling price relatively cheaper. The cause of low-grade quality is that the patchouli oil produced is still containing organic or inorganic colorants that makes the oil dark liquid. Therefore, it is necessary to develop an in expensive means to eliminate colorants in patchouli oil for better quality.

Reference [7] shows, having purified clove oil using 1.2-M H2SO4-activated bentonite, showed that the comparisons of absorbent weight (g) and clove-leave oil volume (ml) were 1:20, 1:40, 1:60, 2:80 and 1:100. His highest absorption outcome was 1:40, meaning that the usage of bentonite was ±2.8% of oil weight.

II. METHODS OF RESEARCH

A. Materials and Instruments

The raw materials used are patchouli oil extracted from farmers’ purification in Rimbo Binuang village, Lingkung Aur District, Simpang Ampek, Pasaman Barat regency.

The chemicals applied for activations of bentonites are bentonite (Al2O3.4SiO2 2H2O); H2SO4 1.2 M, HCL 1.3 M. The chemicals needed for analysis of physical properties in patchouli oil are etanol, dietyl ether, and distilled water.

The instruments used are a flask, two 140-mesh sieves, 100-ml measuring glass, magnetic agitator, screen, porcelain scraper, 230-mesh sieve, water-cash and analytical scales, N-200-type magnesium monel cloth, UV spectrophotometer, water heater conversed at 25°C ± 0.2°C, 5-ml capacitaced pinometer in accordance with available oil volume, furnished with calibrated thermometer, refractometer, and polarimeter.

The experiment was conducted in two stages. The first stage was the activations of bentonites and the second was the purification of patchouli oil. The former was carried out under the guidance of the [7] experiment, supported by in [5]. It was adjusted for the following treatments: inactivated bentonite was sieved by 140-mesh sieve, drawn 300 g and poured into a 2-L flask, and added with 1.2 M H2SO4/HCl at the quantity of 1200 ml (the comparison of 1:4, according to
in [7], agitated by magnetic agitator, activated during 24 hours, screened until it was free of H$_2$SO$_4$/HCl and washed with hot water for perfect detachment of H$_2$SO$_4$ and HCl to reach pH 7, dried up within an oven at temperature 100°C for 1 hour, and scraped by porcelain scraper.

The purification of patchouli oil in this research was under the guidance of clove-oil purification in [30] with the following steps: the patchouli oil of farmer-made purification at the quantity of 200 g was mixed with bentonite in accordance with the treatments of applying shaker bath for 5 hours, deposited for 24 hours, and this oil was separated from bentonite using a centrifuge for 15 minutes, and screened by N-200-type monel cloth.

**B. Analysis of Data**

The Complete Randomized Design (CRD) in factorial was applied. Factor 1 was the activation of bentonites which was done with 2 levels as the followings: inactivated, H$_2$SO$_4$-activated, and HCl-activated. Factor 2 was the application of bentonites with 5 levels: 1%, 2%, 3%, 4% and 5%. Each treatment was repeated twice.

**III. RESULTS AND DISCUSSION**

**A. Observation before Purification**

The observation before purification can be seen in the Table 1.

**TABLE I**

<table>
<thead>
<tr>
<th>No.</th>
<th>Types of Tests</th>
<th>Unit</th>
<th>Before Purification</th>
<th>Requirements</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Clearness (transmittance)</td>
<td>%</td>
<td>69</td>
<td>-</td>
</tr>
<tr>
<td>2</td>
<td>%) Ethanol solubility</td>
<td>-</td>
<td>soluble, pure (Opalesensi ringan), in volume ratio 1:10</td>
<td>Larut, jernih (Opalesensi ringan), in volume ratio 1:10</td>
</tr>
<tr>
<td>3</td>
<td>Total acid</td>
<td>-</td>
<td>6,3112</td>
<td>Max 8</td>
</tr>
<tr>
<td>4</td>
<td>Total ester</td>
<td>-</td>
<td>7,77</td>
<td>Max 20</td>
</tr>
<tr>
<td>5</td>
<td>Alpha copaene (C$<em>{15}$H$</em>{24}$)</td>
<td>%</td>
<td>0,03</td>
<td>Max 0,5</td>
</tr>
<tr>
<td>6</td>
<td>Patchouly alcohol (C$<em>{15}$H$</em>{25}$O)</td>
<td>%</td>
<td>24,74</td>
<td>Min 30</td>
</tr>
<tr>
<td>7</td>
<td>Fe Content % mg/kg</td>
<td>-</td>
<td>19</td>
<td>Max 25</td>
</tr>
</tbody>
</table>

Of the items observed, generally they all have been conforming to SNI 06-2385-2006, except patchouly alcohol.

**B. Observations after Purification**

1) **Clearness (Transmittance Percentage)**

From the Figure 1, it can be seen that the bentonite activations with acids provide higher transmittance percentage than those inactivated for all bentonite applications. According to [6], the downside of such bentonites could be overcome by the process of activations with acids (HCl, H$_2$SO$_4$, and HNO$_3$) to produce the bentonites with higher absorption ability [5].

Transmittance of oil after purification can be seen in fig 1.

![Transmittance of oil after purification](image1)

**Figure 1. The graph of relationship between bentonite activations and bentonite applications and transmittance percentage**

**2) Ethanol solubility**

![Ethanol solubility](image2)

**Fig 2. The graph of relationship between bentonite activation and bentonite application and ethanol solubility of patchouli oil after purification.**

The low values in solubility in H$_2$SO$_4$ activated bentonite with amount of adding 2% in comparing between H$_2$SO$_4$ activated bentonite with HCl activated and non activated bentonite is more active to absorb the waste and with no others substances needed. It proved by the high transmittance and the low iron content in this treatment.

Solubility of the material it influenced by the variation of polarization material itself. More polarization a material towards solvent, more easily material become soluble [1]. Patchouli oil belongs to volatile oil because it content of oxygenated hidrocarbon

**3) Total acid**

Total acid of the oil after purification can be seen in fig 3.

![Total acid](image3)

**Fig 3. The graph of relationship between bentonite activation and bentonite application and total acid.**

The graph 3 showed acid amount of patchouli oil is lower before purification and all the treatments are fulfilled SNI 06-2385-2006. A part of volatile oil is having less amount of free organic acid content that bonded naturally or produced from oxidation process and ester hydrolyze. Oil that has
been dried and protected from light and air is having less amount of free organic acid. The decreasing reaction of acid amount is caused by the existence of acid and alcohol in patchouli oil component above [2]

\[
\text{Carboxylic Acid} \rightarrow \text{Alcohol} \rightarrow \text{Ester} \rightarrow \text{Water}
\]

4) **Total ester**

Total ester of the oil after purification can be seen in Fig 4.

![Graph of relationship between bentonite activation and bentonite application and total ester](image)

Fig 4. The graph of relationship between bentonite activation and bentonite application and total ester.

The increasing of ester amount after purification is caused of the reaction between acid and alcohol formed to ester. This statement is supported by the decreasing of total acid meanwhile the total ester increased but it still fulfilled SNI with maximal 20.

5) **Alpha Copaene (C\textsubscript{15}H\textsubscript{25}) dan Patchouli Alkohol (C\textsubscript{15}H\textsubscript{25}O)**

**TABEL II**

<table>
<thead>
<tr>
<th>No</th>
<th>Analysis</th>
<th>Before Purification</th>
<th>After Purification (Activated Bentonite H\textsubscript{2}SO\textsubscript{4} with 2% applied)</th>
<th>Identification Method</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Alpha copaene</td>
<td>0.03</td>
<td>0.04</td>
<td>GC</td>
</tr>
<tr>
<td>2</td>
<td>Patchouli Alkohol</td>
<td>24.74</td>
<td>25.02</td>
<td>SNI 06-2385-2006</td>
</tr>
</tbody>
</table>

Table I showed that patchouli oil purification is increased alpha copaene and patchouli alcohol. It caused by the absorbing of waste component and color substance then Fe content by bentonite absorbent.

Alpha copaene is required in SNI 06-2385-2006 with max 0.5%, it means the patchouli oil after purification fulfilled SNI 06-2385-2006. Alpha copaene is not really needed in patchouli oil due to this compound is patchoulene which included in class of terpenes that doesn’t give fragrances and alcohol non-soluble [4]

6) **Fe content**

Fe content after purification can be seen in Fig 5.

![Graph of relationship between bentonite activation and bentonite application and Fe content](image)

Fig 5. The graph of relationship between bentonite activation and bentonite application and Fe content.

Fe content of the oil after purification lower than before purification and the lowest is in activated bentonite with H\textsubscript{2}SO\textsubscript{4} that 2% applied, it cause increase % of transmittant value. Sulfit acid is an acid which has equivalent number H\textsuperscript{+} higher than chloride acid therefore is more active to absorb waste and metal oxide in oil.

IV. CONCLUSIONS

The analysis of patchouli oil before purification provider the value of transmitted percentage of 69% and the other physical properties have been eligible to SNI 06-2385-2006, except for the value of patchouli alcohol.

The acid activated bentonit increase the transmittance percentage from 69% to increase (70,80-81,55%). Patchouli alcohol from 24,74% to 25,02% although lower than standard SNI 06-2385-2006. The total acid total ester alpha copaene and Fe content are generally eligible to the SNI.

Further research is necessary to examine the acid concentration for bentonite activation and to know the contact time between bentonite and oil during the purification process. The process of acid washing from bentonite following its activation should use high absorptive ability to absorb faster and more perfect acids.

**REFERENCES**


