

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

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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 Copaene (C₁₅H₂₄) Content, and purity. The method of research used is Complete Randomized Design (CRD) with factorial design. Two factors has been examined are 1) bentonite activations wit 3 levels; inactivated bentonite, H₂SO₄-activated bentonite, and HCl-activated bentonite; and 2) the number of bentonite applications wit 5 levels : 1%, 2%, 3%, 4%, and 5%. And analysis of oil coloris made by using UV spectropotometer 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 copaene content, and patchouli alcohol content. H₂SO₄ –Activated bentonite by 2% given was indicated as te 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 copaene 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, *Lingkungan 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 spectrovotometer, water heater conversed at 25⁰C ± 0.2⁰C, 5-ml capacitated picnometer 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_2SO_4/HCl and washed with hot water for perfect detachment of H_2SO_4 and HCl to reach pH 7, dried up within an oven at temperature $100^{\circ}C$ 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_2SO_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
THE RESULT OF PATCHOULI –OIL ANALYSIS BEFORE PURIFICATION AND GRADING REQUIREMENTS OF SNI 06-2385-2006

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

Of the items observed, generally they all have been conforming to SNI 06-2385-2006, except patchouli 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_2SO_4 , and HNO_3) to produce the bentonites with higher absorption ability [5].

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

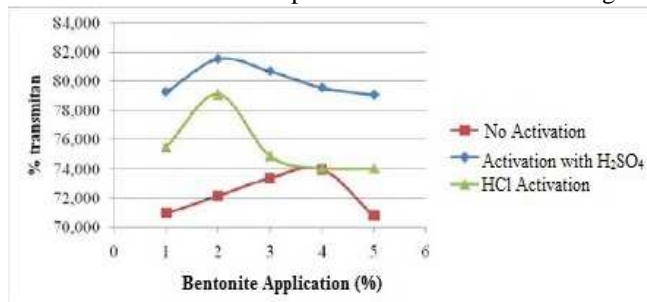


Figure 1. The graph of relationship between bentonite activations and bentonite applications and transmittance percentage

2) Ethanol solubility

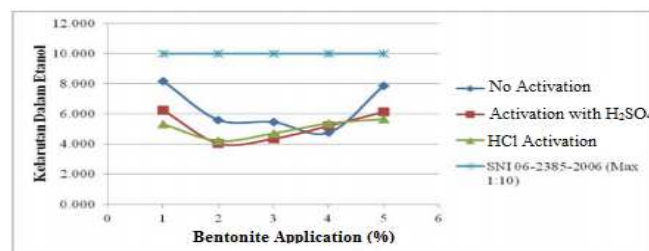


Fig 2 . The graph of relationship between bentonite activation and bentonite application and ethanol solubility of patchouli opil after purification..

The low values in solubility in H_2SO_4 activated bentonite with amount of adding 2% in comparing between H_2SO_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 hydrocarbon

3) Total acid

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

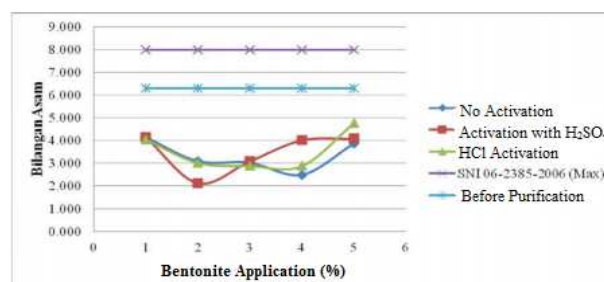
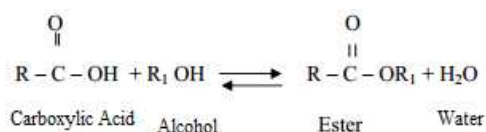


Fig 3 The graph of relationship between bentonite activation and bentonite application and total acid.

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]



4) Total ester

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

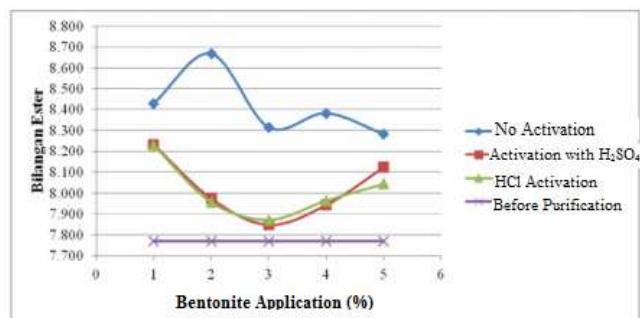


Fig 4. The graph of relationship between bentonite activation and bentonite application and total ester.

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 ($C_{15}H_{25}$) dan Patchouli Alkohol ($C_{15}H_{25}O$)

TABEL II

THE RESULT OF ALPHA COPAENE AND PATCHOULI ALCOHOL BEFORE AND AFTER PURIFICATION (THE BEST TREATMENT ONLY)

No	Analysis	Result		Identificati on Method
		Before Purifica tion	After Purification (Activated Bentonite H_2SO_4 with 2% applied)	
1.	Alpha copaene (%)	0,03	0,04	GC
2	Patchouli Alkohol (%)	24,74	25,02	SNI 06-2385-2006

Table 1 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 wth 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 conten after purification can be seen in fig 5.

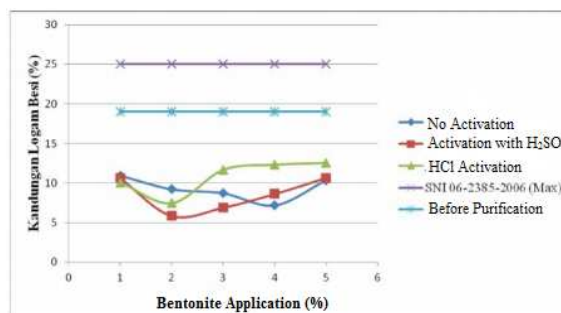


Fig 5. The graph of relationship between bentonite activation and bentonite application and Fe content

Fe content of the oil after purification lower than before purification and the lowest is in activated bentonite with H_2SO_4 that 2% applied, it cause increase % of transmittant value. Sulfit acid is an acid which has equivalent number H^+ 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.

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