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Enhancing the Quality of Patchouli Oil using Zeolite and Bentonite Nano-Particle Adsorbents

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Abstract

The overall quality of patchouli oil is closely related to the purification process during manufacturing. This research provided an effective and efficient method for purifying Aceh patchouli oil to secure high-quality patchouli oil. Purification was done by utilizing natural mineral sources that were sufficiently available in Aceh and applying them in various adsorption processes, analyzing the adsorption of Fe levels in patchouli oil, and characterizing nano adsorbents used in the adsorption process. Nanoparticles of zeolite and bentonite were used during the adsorption process with patchouli oil sourced from the South Aceh area. The adsorption process purified 20 mL of patchouli oil at a temperature of 45 ℃ with variable mass nano adsorbent 1; 3; 5 g, and variable stirring time 60; 180; 300 minutes. The purification filtrate was later analyzed for patchouli alcohol levels using Gas Chromatography-Mass Spectrometry (GC-MS) and Fe levels using UV-Vis Spectrophotometry. The characterization of nano adsorbents was conducted using Scanning Electron Microscopy (SEM) and X-ray Diffractometry (XRD). The results showed that the most efficient adsorption process occurred in nano zeolite with a mass of 5 g and a stirring time of 300 minutes, where the patchouli alcohol content was 28.54%, Fe 0.8165 mg/kg. Meanwhile, nano zeolite's most significant constituent components were silicon oxide (SiO_2) of 48.9% and silicon carbide (CSi) of 31.3%. Visually the sample did not change color due to the low quality of patchouli oil, but the Fe level in the patchouli oil samples complied with SNI 06-2385-2006 with a maximum of 25 mg/kg. In addition, nano zeolite had an adsorption capacity of 0.96 mg/g with an adsorption efficiency of 98.49%.

1. Introduction

Indonesia is the world's largest supplier of patchouli oil, meeting 90% of the world's patchouli oil needs and Aceh Province contributing as much as 70%. *Patchouli oil* is an essential oil obtained by distilling leaves, stems, and branches from the patchouli plant. Indonesian patchouli oil is regularly exported internationally to France, Singapore, the United States, the United Kingdom, Germany, India, Spain, and the Netherlands as raw materials for fragrance oils, cosmetics, pharmaceuticals, and other industries [1].

The main component of patchouli is *patchouli alcohol* (PA), a compound belonging to the sesquiterpene group with the molecular formula $C_{15}H_{26}O$. The higher the PA level of the patchouli oil, the better the quality of the oil itself. There are various strains of Aceh patchouli seedlings, including Sidikalang varieties with a PA rate of 37.3%, Tapak Tuan with a PA rate of 35%, Cisaroni with a PA rate of 33.1%, and Lhokseumawe with a PA rate of 30.5% [2]. *Patchouli alcohol* also has properties that serve as a binding aroma (fixation) so that fragrances last longer.

The farmers and industry players generally carry patchouli oil's cultivation and production process in Indonesia using conventional technology. There is limited knowledge and research around the patchouli oil extraction process. This negatively impacts the quality of patchouli oil products. Most patchouli oil is produced through an outdated distillation process that uses distillation kettles made from used drums with river water as a solvent, making the final patchouli oil color dark and murky.

Natural minerals reduce and eliminate these impurities in an adsorption process. One of the methods used by researchers to reduce impurities or waste is the adsorption method. Adsorption is a process caused by fluid accumulation on a liquid or solids surface and forms a solid-liquid boundary. Adsorption has two types of procedures: physical adsorption and chemical adsorption. The adsorption process is influenced by several factors, such as stirring speed, surface area, the types of adsorbents, pH, and temperature [3].

This study used the natural adsorbents zeolite and bentonite, modified into nanoparticles, in the adsorption process. According to Emelda *et al.* [4], several physical and chemical modifications can improve the adsorption ability of zeolite. Bentonite is a hydrated alumina silicate mineral included in phyllosilicates or layered silicates consisting of a tetrahedral network SiO_4 [5]. Bentonite has cation-anion that can be distributed a layered structure that can expand or swell. Bentonite can also be used in the adsorption process but has limited adsorption capability. However, activating strong acid solutions, such as HCl, H_2SO_4, and HNO_3, results in bentonite with higher adsorption capability. Bentonite activation usually requires an acid solution, resulting in bentonite with a larger active site, greater surface acidity, and increased adsorption capacity [6].

Based on the potential of zeolite and bentonite that have been described, it is necessary to know more about the adsorption process with nano adsorbents to obtain patchouli oil with Fe^{2+} metal ion content that meets SNI 06-2385-2006 standards. The aims of this study were 1. Characterize nano bentonite and nano zeolite as adsorbents, 2. Analyze the levels of *patchouli alcohol* and Fe metal ions contained in Aceh patchouli oil before and after the adsorption process. 3) Analyze the adsorption power of nano bentonite and nano zeolite in the process of purification of Aceh patchouli oil, 4) Analyze the influence of treated test parameters on the purification process of Aceh's patchouli, and 5) Determine the optimum parameters to improve the quality of Aceh patchouli oil.

2. Materials and Research Methods

This research was conducted in the Laboratory of Operations and Processes Unit, Material Analysis Laboratory of The Department of Chemical Engineering, and Essential Laboratory Research Center (ARC) PUI PT Nilam Aceh, Syiah Kuala University, Darussalam, Banda Aceh, from January–May 2020.

2.1. Materials and Tools

The ingredients used in this study were as follows: Subulussalam patchouli oil samples, Lhouksemawe natural zeolite, Lhouksemawe natural bentonite, distilled water, Ethanol 96% (MERCK, Reagent Ph Eur.), HCl, and HNO_3.

The tools used in this study were as follows: a stirrer plate (Fisher Scientific), beakers 100 mL and 5 mL (Pyrex), an Erlenmeyer 250 mL (Pyrex), measuring cups 10 mL and 100 mL (Pyrex), measuring flasks 100 mL and 250 mL (Pyrex), tweezers, analytical scales (Mettler Toledo), a test tube, a GC-MS Shimadzu type GCMS-QP2010, a spectrophotometer UV-Vis (Shimadzu 1800), a Ball Mill FRITSCH, a Scanning Electron Microscopy (SEM) JOEL Type JSM-6701F, X-ray Diffractometry (XRD), a test tube (Pyrex), and a refractometer and the 400 mesh (Macros Testing Sieve).

2.2. Research Design

In this study, each nano adsorbent was characterized using SEM and XRD. Iron content was analyzed using UV-Vis Spectrophotometry. Patchouli oil components were analyzed using GC-MS. The optimum parameter of patchouli oil production was determined based on the iron content following the standard of SNI 06-2385-2006.

2.3. Research Procedure

The adsorption experiments were conducted using a stirred reactor with bentonite and nano zeolite with variations in the number of adsorbents, contact time, and temperature. The adsorption process can be seen in the diagram in Figure 1.

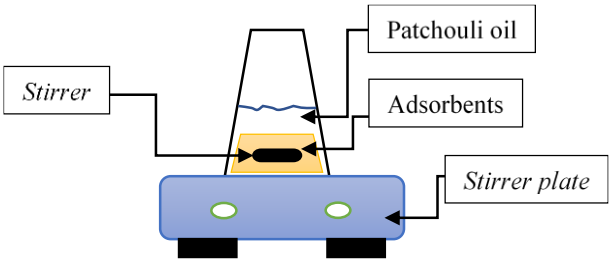

Figure 1. Adsorption process using adsorbents with patchouli oil samples

2.3.1. Activation and calcination process of adsorbents

The adsorbents used in this study were dry. The adsorbents were crushed into nanoparticles using *Ball Milling* for 26 hours to reduce their size. After passing through a 400-mesh sieve, the resulting nano adsorbent underwent a chemical activation process using 37% HCl for 1 hour. They were then dried using the oven to a temperature of 100 °C. Next, the activated nano adsorbents were calcified using *a furnace* at 600 °C for 6 hours. Finally, the activated nano adsorbents were characterized using XRD, and the surface structure was analyzed using SEM.

A total of 2–6 g of nano adsorbent in the form of dry powder was put into a prepared glass to determine the crystal structure and composition of the elements contained in the sample. X-ray diffraction graphs are

generated through X-rays, which at a certain angle, scan crystals with a particular atomic distance. The width of the peak provides information on the crystal size, while the intensity of the XRD peak indicates the magnitude of the X-ray diffraction. X-rays are then fired at the sample at a certain angle through a *divergent slit*, then diffracted and subjected to constructive interference. This diffraction X-ray is then passed through the *receiver slit* and captured by the detector. XRD analysis results in the form of a relationship between 2Θ (diffraction angle), d (thickness of the unit cell or distance between crystal planes), and I (diffraction light intensity) in the form of the appearance of several peaks.

The result of adsorbent pore volume value is obtained by using Bragg equation is as follows:

$$d = \frac{\lambda}{2 \sin \theta}$$

Description:
d = pore volume (nm)
λ = Wavelength (nm)
θ = interpolation distance

2.3.2. Determination of optimum stirring mass and time

Determination of the mass and optimum stirring time was carried out through the batch method. Patchouli oil must be characterized using GC-MS to analyze patchouli alcohol content. After that, 100 mL of sample was inserted into a beaker. Nano adsorbents were added to the sample with mass variations of 1 gram, 3 grams, and 5 grams. The stirring process used a magnetic stirrer with a stirring speed of 100 rpm with a time variation of 1 hour, 3 hours, and 6 hours. The sample was then filtered to obtain the filtrate for characterization using GC-MS and UV-Vis Spectrophotometry. While GC-MS was turning on, the 10 mL of desorbed patchouli oil was prepared. Once the instrument was ready, the sample was injected with a specific volume. The sample will be analyzed, and when the analysis time ended, chromatograms of constituent compounds in the sample will be produced.

The Fe content in the sample was analyzed by measuring the absorbance value of Fe(III) standard solution with the addition of sodium thiosulfate. Measurement was using UV-Vis spectrophotometry at a wavelength of 450–560 nm. Then, the sample solution with a concentration range from 0 to 3.5 ppm was made to be analyzed using a UV-Vis spectrophotometer to obtain the maximum wavelength. Next, a calibration curve was formed between adsorption and tilapia oil samples.

2.4. Test Parameter Analysis

The patchouli oil used in the adsorption process underwent tests to determine color and refractive index characteristics. The test was done before the adsorption process and after adsorption. The parameter test was reviewed by comparing the quality of patchouli oil following SNI 06-2385-2006 (Table 1).

Table 1. Indonesian Value Standard result of quality analysis of patchouli oil SNI 06-2385-2006 [7]

No	Characteristics	Description
1	Color	Light yellow to reddish-brown
2	Density, 25 °C	0.95-0.975
3	Bias index, 20 °C	1.507-1.515
4	Solubility in ethanol 90%, 25-30 °C	Soluble - clear
5	Acid number, max.	5.0
6	Patchouli alcohol ($C_{15}H_{26}O$)	Min 30%
7	Alpha-copaene ($C_{15}H_{24}$)	Max 0.5%
8	Iron content (mg/kg)	Max 25 mg/kg

2.4.1. Type Weight Test

The clean and dry, empty Pycnometer tool was weighed, then filled with water, and put in a thermostat at a temperature of 28 °C for 15 minutes. The pycnometer was removed and dried at room temperature, then drained. This process was then repeated with patchouli oil instead of water.

2.4.2. Bias index

Alcohol was used to clean the prism on the reflectometer, and then the oil was dripped onto the prism using a dropping pipette. The prism was aligned and arranged on the slide to get a clear line between light and dark. As for reading the refractive index, the switch was set to a narrow boundary line with the intersection of two intersecting lines.

2.4.3. Solubility in ethanol 96%

A total of 1 mL of patchouli oil was inserted into a 10 mL measuring glass, 96% ethanol was added in 5 increments, and the burette was shaken until homogeneous. Each addition of 0.5 mL of 96% ethanol to the burette was shaken, and solubility properties were observed until the 10 mL ethanol addition limit was reached.

2.4.4. Sour numbers

In this analysis, 2.5 g of oil was inserted into a 100 mL condensing flask. Then 15 mL of ethanol 96% was added along with 3 drops of 1% phenolphthalein solution. A standard solution of NaOH 0.01 N was used for the administration of free acids. The addition of alkali drops during titration was approximately 30 drops per minute. The contents of the pumpkin should be shaken continuously during titration. When a red color appeared and did not disappear in 10 seconds, this indicated the titration endpoint.

3. Results and Discussion

3.1. Adsorbent Characteristic

3.1.1. The Characterization with *X-ray Diffractometry* (XRD)

The XRD test results from zeolite and bentonite adsorbents can be seen in Figure 2 and 3. Figure 2 describes the content contained in zeolite adsorbent material that contains 45% *Silicon Oxide* (SiO_2); 27.2% $Na(Si_2O_4(OH))$; 25.2% *Carbon* (C); 1.9% *Silicon Sulfide* (SiS_2); 0.7% *Sodium tecto-alumosilicate hydrate*

($Na_6Al_6Si_{10}O_{32}$); and 5.1% of unidentified material. At the peak of the main zeolite adsorbent, the XRD identification result was 2θ = 26.54° (d=3.3547Å) with a pore volume of 0.11857 nm. While in Figure 3 the content contained in bentonite adsorbent material that contains 48.9% *Silicon Oxide* (SiO_2); 31.1% *Silicon Carbide* (CSi); 9.8% *Dilithium Sulfate* (Li_2O_4S); 5.8% *Cadmium Cyanide* ($Cd(CN)_2$); 4.2% *Calcium Oxide* (CaO); and 2.1% of unidentified material. The result of XRD identification at the main peak of bentonite adsorbents was 2θ = 26.5775° (d=3.35119 Å) with a pore volume of 0.116602 nm.

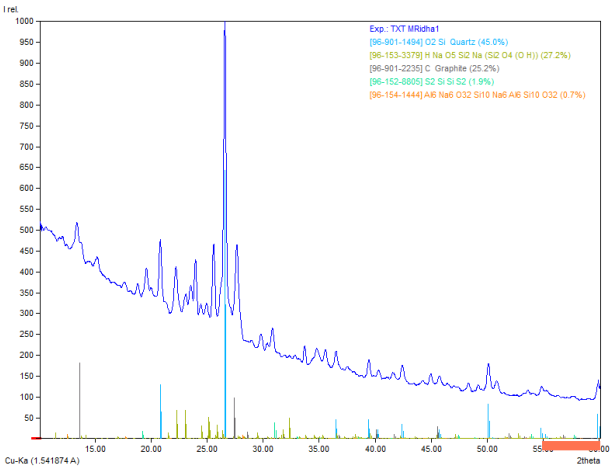

Figure 2. Zeolite characteristic test results using XRD

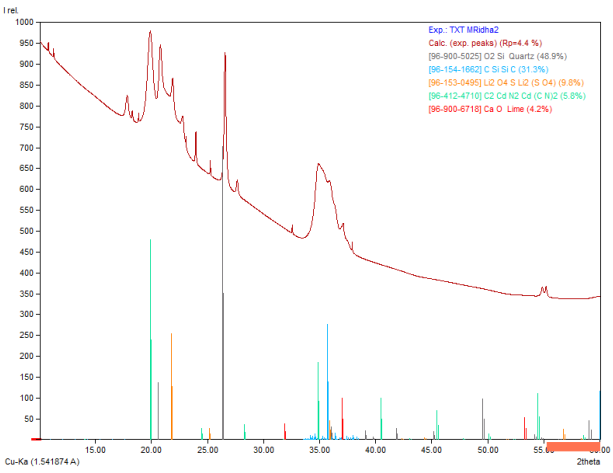

Figure 3. Bentonite characteristic test results using XRD

3.1.2. The characterization using *Scanning Electron Microscopy* (SEM)

Morphological size characteristics test and grain distribution on each nano adsorbent were conducted using SEM magnification 20,000 times. Figure 4 and 5 highlight the nano adsorbent zeolite and bentonite morphology. Through SEM, we can determine the characterization of the nano zeolite morphology and see that it has been activated. According to Figure 4, it can be observed that zeolite was successfully scaled down to nanoparticles.

The Bentonite characterization using SEM also determined the morphology of the activated nano bentonite. Based on Figure 5, the bentonite was successfully reduced in size to nanoparticles. The analysis of the two samples showed that the sample has nano-sized particles that have size of <1 µm. Nanoparticles are particles that have particle measurements with a diameter of 1–1000 nm or 0.001–1 µm [8].

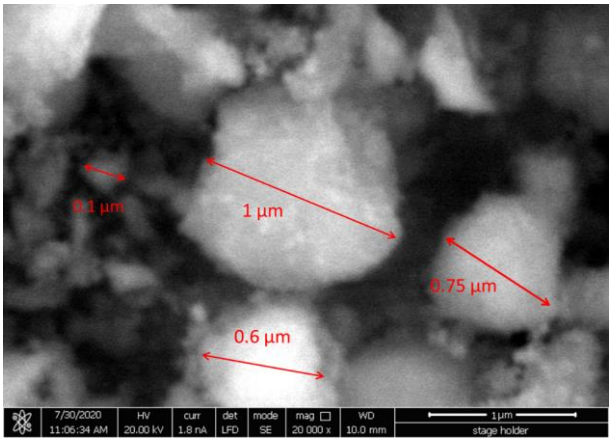

Figure 4. Zeolite characteristic test results using SEM

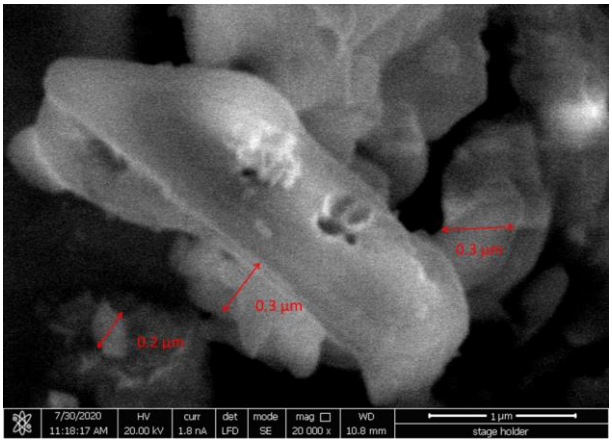

Figure 5. SEM image of bentonite

3.2. Fe Metal Content in Patchouli Oil

Other parameters such as its metal content, especially iron (Fe), were also tested to assess the quality of patchouli oil. According to the SNI 2006, the patchouli oil must contain Fe levels at or lower than 25 mg/kg. High iron content in patchouli oil caused the color of patchouli oil to turn reddish-brown. Iron ions dissolved in patchouli oil were derived from iron flutes and water used during the oil refining process.

The iron metal content in patchouli oil and the calibration curve in the raw solution can be seen in Figure 6. Results of calibration curve at concentration 0; 0.5; 1; 1.5; 2; 2.5 and 3 mg/kg obtained absorbance values of 0; 0.286; 0.516; 0.74; 0.981; 1.178 and 1.356 nm, respectively. So that the linear line equation of the relationship between adsorption and concentration is obtained where y = 0.2256x – 0.18 and R^2 = 0.9958. Where y is adsorption value and x is concentration, while R^2 is relationship between adsorption and concentration. The calibration curve showed accuracy in the formation of concentrations of 99.58% at R^2 value of 0.9958.

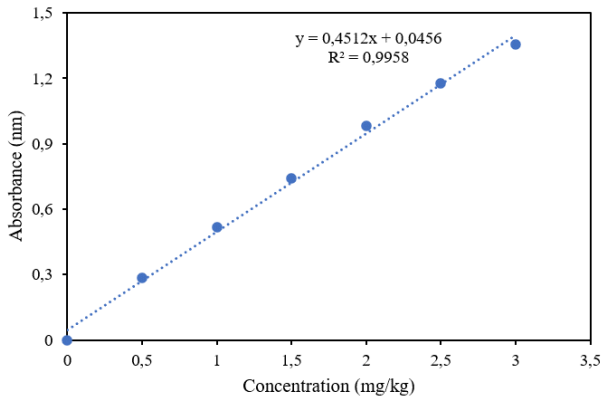

Figure 6. Calibration curve between absorbance and concentration on the raw solution

3.2.1. Preliminary Test in Optimum Mass Determination of Zeolite and Bentonite Adsorbents with The Addition of Nano-Adsorbents

This preliminary test was conducted to determine the optimum mass of adsorbents in adsorbing Fe metals. The relationship between zeolite and bentonite adsorbent masses with Fe metal content can be seen in Figure 7. Comparison between zeolite and bentonite adsorbents with the addition of nano adsorbents in adsorbing Fe metals. Results showed that the amount of Fe sorbed by 1 gram of zeolite was 12.78 mg/kg, while in bentonite with the same mass, Fe levels obtained were greater by 19.99 mg/kg. The most decrease in Fe metal is shown in the adsorbents with a mass of 5 g, 0.816 mg/kg for zeolite, and 8.02 mg/kg for bentonite. The occurrence of increased adsorption in the adsorbate using adsorbents. Therefore, the optimum mass was obtained in the Fe metal adsorption process using zeolite and bentonite adsorbents at a mass of 5 g nano adsorbents.

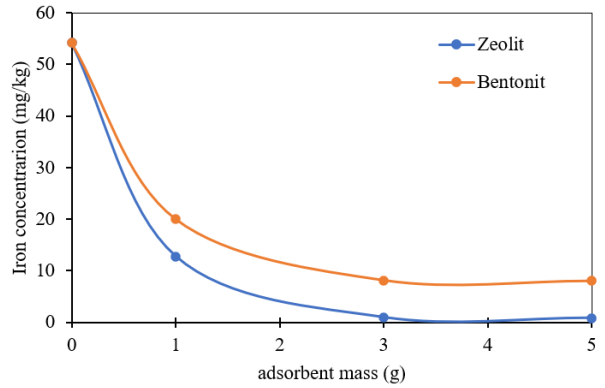

Figure 7. The relationship between adsorbent mass and Fe metal content at 300 minutes contact time and stirring speed of 100 rpm

The concentration differences of the Fe metal with the adsorbents that happened in the solvent were enormous, or the driving force is considerable. Thus, the mass transfer process of Fe metal contained in the patchouli oil's solvent to the patchouli oil's adsorbents occurred quickly. The ability of adsorbents to adsorb very large adsorbates is one of the other factors that affect it. At the beginning of the adsorption process, Fe metal was adsorbed very essentially. It was suspected that the active site of the adsorbent is still a lot. As time increased, the smaller the mass transfer rate would eventually be constant. The small amount of driving force triggers a difference in the concentration of Fe metal in the solvent and the adsorbent so that its adsorption capability was small [4].

3.2.2. The Effect of Contact Time on Fe Metal Levels with Zeolite and Bentonite Adsorbents with Nano-Adsorbent Addition

The contact time between adsorbents and adsorbates affected the decrease in Fe metal levels (Figure 8). In this study, the variation in the contact time was 60, 180, and 300 minutes. The more extended contact time can see the adsorption process with zeolite adsorbents with nano adsorbents. The results showed that with increasing contact time from 60 to 300 minutes, the Fe removal decreased, namely 38.21; 24.26; and 0.816 mg/kg, respectively. This indicated that decreased concentration from an initial concentration of 38.21 mg/kg to 0.816 mg/kg. However, within 180 minutes, the Fe metal levels decreased only slightly, and the decline was very significant up to the 300 minutes.

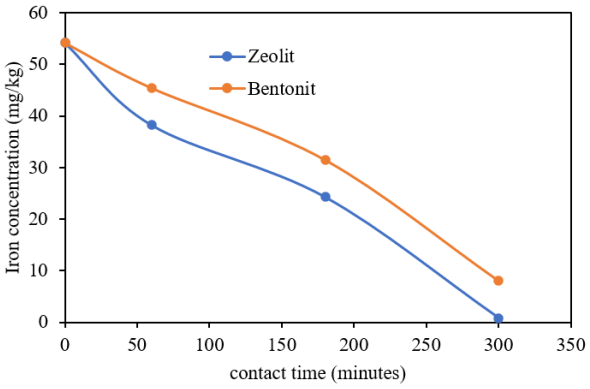

Figure 8. Relationship between contact time and Fe metal content at optimum adsorbent mass and stirring speed of 100 rpm

As for bentonite adsorbents with the addition of nano adsorbents, from the picture can be seen a decrease in the concentration of metal Fe, at a contact time of 60 minutes, obtained Fe metal concentration of 45.42 mg/kg and up to a contact time of 180 minutes continue to decrease relatively slightly by 31.476mg/kg. Meanwhile, at 300 minutes, Fe metal concentration showed a significant decrease in 8.02 mg/kg value.

The reduced concentration of Fe metal in patchouli oil is a symptom of the adsorption process, where several metals are adsorbed on the surface of zeolite and bentonite adsorbents (Figure 8). Patchouli oil containing a large enough iron content from the distillation using drums is taken for analysis of Fe content reduction using adsorption method with zeolite and bentonite in nanoparticle size. After the adsorption process using nano zeolite with stirring rotation 100 rpm for 300 minutes with a mass of 5 grams can lower iron content by 53.35 mg/kg (98.49 %), and the remaining Fe content

in the sample was 0.816 mg/kg. While the adsorption process using nano bentonite under the same conditions can lower Fe levels by 46.14 mg/kg (85.18%), and the remaining Fe content in the sample was 8.02 mg/kg.

Based on these results, it can be concluded that zeolite adsorbents are more effective than bentonite in adsorbing Fe metal in patchouli oil. This is influenced by the number of nanoparticles and adsorption capacity in adsorbents—the greater the adsorption capacity in adsorbents, the greater Fe levels in the sample [9].

3.3. Adsorption Results in Optimum Conditions

3.3.1. Effect of Mass Optimum Contact Time on Adsorption Capacity

Based on the test results obtained, the optimum mass of the adsorption process is at a mass of 5 g adsorbent. Then, the optimum mass was carried out by the adsorption process by varying the contact time 60, 180, and 300 minutes (Figure 9). The adsorption capacity was using nano zeolite. In Figure 9, the relationship of the absorbance time with adsorption capacity in zeolite adsorbents with the addition of nanoparticles in it and obtained line equations from linearization is y = 0.0031x + 0.0359 with R^2 = 0.9834. The adsorption capacity at the time of contact 0; 60; 180; and 300 in a row of 0; 0.2872; 0.5382; and 0.9657 mg/g. At times above 300 minutes, it was estimated that zeolite adsorbents with nanoparticles would reach equilibrium. Equilibrium time is when there is no longer the adsorption process of substances to the adsorbent medium. The minimum adsorption capacity was 60 minutes with an adsorption capacity of 0.28728 mg/g, while the maximum capacity is 0.965763 mg/g.

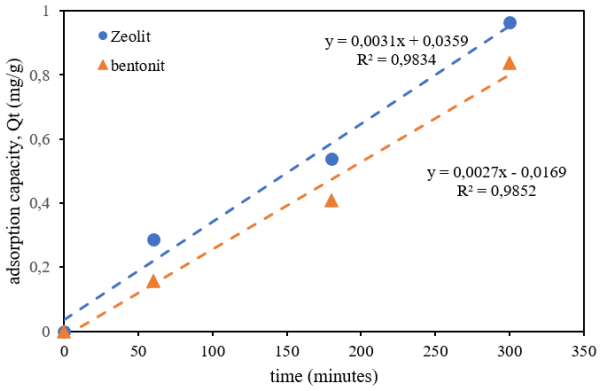

Figure 9. The relationship of contact time changes to adsorption capacity using zeolite and bentonite nano adsorbent with an additional 5 g

The next step is the additional adsorption capacity using bentonite with nanoparticle content. The relationship between adsorption time and adsorption capacity on bentonite adsorbents can be seen with the addition of nanoparticles in it. The equation of the linear line is y = 0.0027x – 0.0169 with R^2 = 0.9852. From the test results obtained adsorption capacity at the time of contact 0; 60; 180; and 300 in a row of 0; 0.1574; 0.40849; and 0.83596 mg/g. It can be estimated that at times above 300 minutes, bentonite adsorbents with nanoparticles will reach equilibrium time.

The longer the contact time in the adsorption process, the greater the value of adsorption capacity. Increasing the contact time between adsorbent and adsorbate was increased the amount of the substance adsorbed [10].

3.3.2. Effect of Optimum Conditions Contact Time on Adsorption Efficiency

The adsorption efficiency is also calculated at optimum conditions, i.e., at an adsorbent mass of 5 grams. The efficiency of adsorption of Fe metal using nano zeolite can be seen in Figure 10. The relationship of adsorption time with adsorption efficiency in zeolite adsorption with nanoparticles in it and obtained line equations from linearization is y = 0.3112x + 3.7755 with R^2 = 0.9835. The test results obtained efficiency at contact time of 0; 60; 180; and 300 in a row of 0; 29.46; 55.20; and 98.49%. The minimum efficiency was at 60 minutes with an adsorption capacity of 29.46%, while the maximum efficiency was 98.49%.

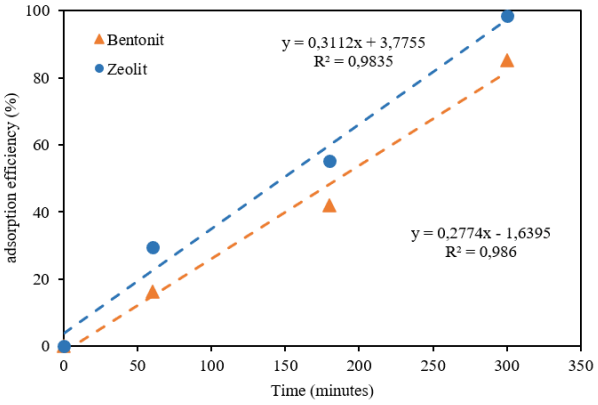

Figure 10. Relationship of contact time to adsorption efficiency with the addition of nano zeolite and bentonite

Furthermore, in Figure 10, the relationship of absorbance time with adsorption efficiency in bentonite adsorbent with the addition of nanoparticles and obtained line equations from linearization is y = 0.2774x – 1.6395 with R^2 = 0.986. It obtained efficiency at the time of contact 0; 60; 180; and 300 in a row of 0; 16.15; 41.86; and 85.18%. The minimum efficiency was at 60 minutes with an adsorption capacity of 16.15%, while the maximum efficiency was 85.18%. The longer the contact time in the adsorption process, the greater the efficiency value of adsorption. This was influenced by the activated adsorbents that can improve adsorption power in adsorbing Fe metal ions in patchouli oil samples [11].

3.3.3. Kinetics Adsorption Pseudo First Order and Second-Order Reactions

A reaction whose speed depends on only one of the substances reacting is a pseudo-first-order reaction proportional to one of the reactant ranks. Based on Figure 11, the line equation obtained from the linearization is y = -0.0051x + 2.4652 with R^2 = 0.9538. A k_1 value was 2.4652 1/min, and Q_e value of equation (theoretical Q_e) was 0.994 mg/g.

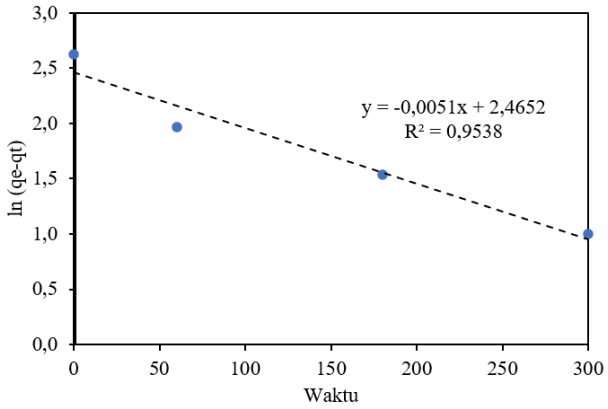

Figure 11. Kinetics adsorption pseudo-first-order contact time relationship to ln (qe-qt)

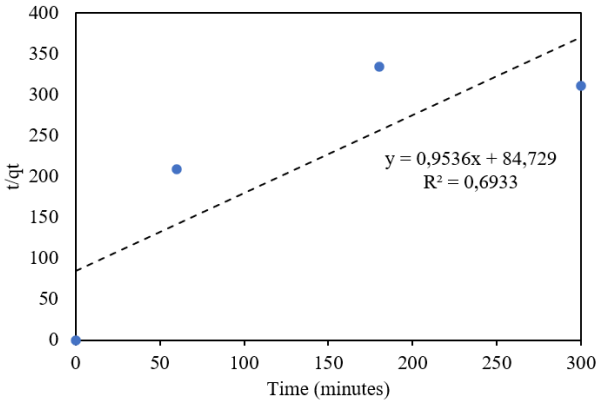

Figure 12. Kinetics adsorption pseudo-second-order contact time relationship to t/qt

The pseudo kinetics of the second order is obtained by graphing the relationship between contact time (t) against t/qt. The pseudo-order kinetic graph 2 is shown in Figure 12 with an R^2 value of 0.6933. A reaction whose speed is directly proportional to the concentration of two reactants or directly proportional to the square of the concentration of one of the reactants is called a second-order reaction. The line equation of linearization is y = 0.9536x + 84.729 with R^2 = 0.6933. The equation of line has a Q_e value of the equation (theoretical with R^2 Q_e) of 1,048 mg/g, and the value of k_2 is 0.8671 l/min. By comparing the regression value (R^2) closest to 1 between the two adsorption kinetics, the adsorption process in this study followed the pseudo kinetics first order, namely with R^2 = 0.9538, which explains the adsorption process follows adsorption physically. Adsorption that occurs due to van der Waals forces is known as physics adsorption. Van der Waals forces are a relatively weak pull-pull force between adsorbate and adsorbent surfaces. This force is strongly influenced by adsorption capacity on the surface of adsorbents.

3.4. Comparison of Optimum Condition Adsorption Results with Distillation Patchouli Oil Using Drums

From the adsorption data obtained at the optimum condition with drum-distilled patchouli oil analyzed with Chromatography Gas, the Kg-SM analysis of essential oil components of patchouli plants can be obtained through a chromatogram. Based on Figure 13, as many as five main types of compounds that comprise patchouli oil can be seen from the top of the highest line in the picture. The components of patchouli oil can be seen in Table 2. Based on the data of patchouli oil composition from the chromatography test, it is clear that the area of patchouli alcohol contained in patchouli oil from drums before and after adsorbing using zeolite in a row is 24.28% and 28.54%, respectively (Figure 13 and 14). The concentration indicated the purity level of the footage analyzed. The patchouli alcohol fraction analysis results after adsorption showed a retention time of 16.960, with a patchouli alcohol content of 28.54%.

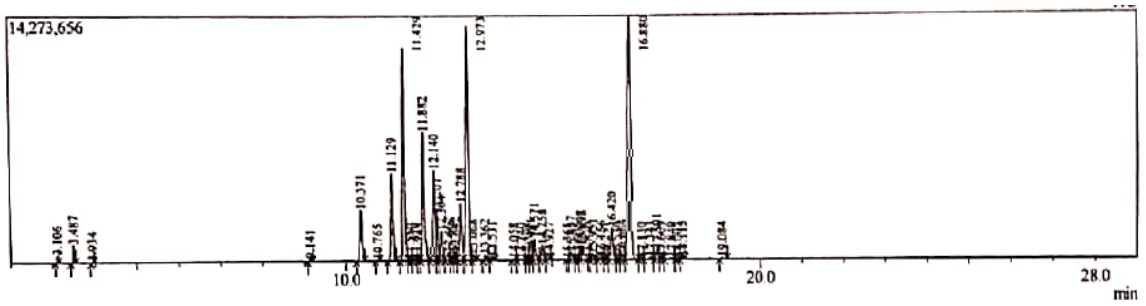

Figure 13. Distilled patchouli oil chromatogram using drums before adsorption

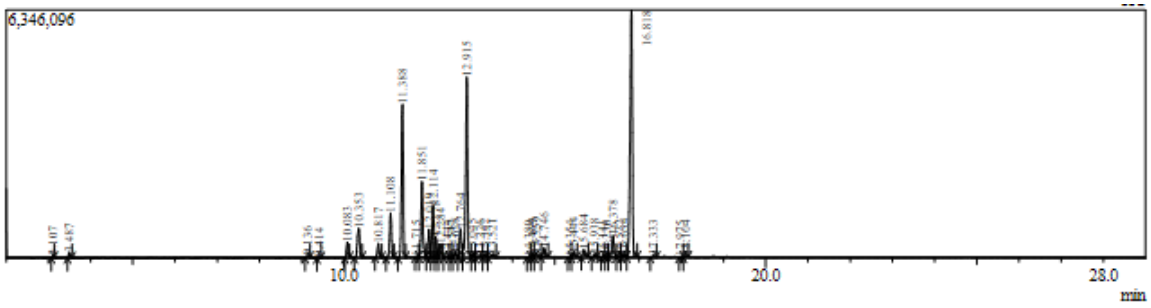

Figure 14. Distilled patchouli oil chromatograms using drums after adsorption of optimum conditions with nano zeolite

Table 2. Constituent components of patchouli oil

No	Chemical composition	Patchouli oil before adsorption	Patchouli oil after adsorption
1	Caryophyllene	0.04	0.04
2	Alpa-Patchoulene	5.58	4.99
3	Alpa-Guene	14.21	13.66
4	Patchouli Alcohol	24.28	28.54
5	Seychellene	8.28	7.56

Patchouli has a relatively high boiling point, as evidenced by the appearance of a peak at the end of the patchouli oil chromatogram. The relatively high boiling point causes patchouli oil to have fixative properties. Fixative properties are known as binders of other essential compounds, so if mixed with patchouli oil, then the boiling point of a relatively low essential compound will rise. The high boiling point of this mixture causes the aroma of essential oils if the mixture is not volatile. The unique properties of patchouli oil are most noticeable in Aceh Patchouli. These properties are used as fragrance binders in perfume or cosmetic products [12].

This study shows that an adsorption method using nano zeolite particles can adsorb 98.49% of the Fe content in patchouli oil. The area of the main component of patchouli oil is larger. The results of the gas chromatography also showed various constituent components of patchouli oil, in addition to patchouli alcohol. Five components are subject to fundamental changes in either decreasing or increasing presentation area, as stated in Table 2. Based on the analysis results, the characteristics between patchouli oil after adsorption at optimum conditions with patchouli oil distilled using drums can be seen in Table 3, based on SNI 06-2385-2006.

Table 3. Specification Comparison based on SNI 06-2385-2006

No	Characteristics	Before adsorption	After adsorption
1	Color	Reddish Brown	Reddish Brown
2	Density, 25 ℃	0.97	0.90
3	Bias index, 20 ℃	1.491	1.509
4	Solubility in ethanol 96%, 25-30 ℃	Insoluble yellow	Insoluble yellow
5	Acid number, max.	10 mL	6.0 mL
6	Patchouli alcohol ($C_{15}H_{26}O$)	24.28 %	28.54%
7	Alpha-copaene ($C_{15}H_{24}$)	-%	1.25 %
8	Iron content (mg/kg)	54.17 mg/kg	0.8165 mg/kg

4. Conclusion

Based on the research that has been done, it can be concluded as follows: Fe^{2+} levels in patchouli oil decreased in concentration under optimum conditions at the adsorbent mass of 5 g. Adsorption capacity in zeolite adsorbents at 60 minutes has a minimum adsorption capacity of 0.28728 mg/g, while the maximum capacity is 0.965763 mg/g. Fe^{2+} levels in patchouli oil decreased in concentration under optimum conditions at a contact time of 300 minutes. *Patchouli alcohol* content of patchouli oil from used drums increased adsorption from 24.28 % to 28.54 % after desorption. The Fe metal in the initial patchouli oil amounted to 54.17 mg/kg, after desorption within 300 minutes with zeolite adsorbents decreasing to 0.8165 mg/kg (98.49%) and bentonite amounting to 8.0275 mg/kg (85.18%). Adsorption kinetics in this study followed order equation 1 with a value of R^2 of 0.9538.

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