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# Effect of Bayah natural zeolite for purification of waste cooking oil as feedstock of alkyd resin

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ABSTRACT

The waste of cooking oil was increased due to the cooking oil consumption increases. Waste cooking oil can reuse for raw material after the adsorption process with Bayah's natural zeolite, such as feedstock of alkyd resins. This paper studied the effect of the mass of Bayah natural zeolite (w/w %) on the characteristics of waste cooking oil. The Bayah natural Zeolite as adsorbent to reduce the value of free fatty acids and peroxide numbers The first stage is the preparation of waste cooking oil and Bayah natural zeolite, and then activation of Bayah natural zeolite, and the adsorption process of waste cooking oil with 15 and 30% w/w of zeolite. The results show the characteristics of waste cooking oil which before being adsorbed had an acid value of 1.51% and a peroxide value of 28.43 mek $O_2$/kg which had passed the quality standard parameters of cooking oil quality. The effect of alkaline (NaOH 15 and 30%) activated zeolite to waste cooking oil decreases the free fatty acid content to 0.98% and 0.75%, and the peroxide value was 19.54 mek $O_2$/kg and 18.38 mek $O_2$/kg, respectively. The greater the amount of zeolite added the lower the acid number and peroxide value in the oil, thereby increasing the quality of the oil as raw material for alkyd resins.

ABSTRAK

Minyak jelantah mengalami kenaikan seiring dengan meningkatkanya konsumsi minyak goreng. Minyak jelantah dapat digunakan kembali untuk bahan baku setelah proses adsorpsi dengan zeolit alam Bayah, seperti untuk bahan baku alkyd resin. Makalah ini mempelajari pengaruh massa zeolit alam Bayah (b/b %) terhadap karakteristik limbah minyak jelantah. Tahap pertama adalah preparasi minyak jelantah dan zeolit alam Bayah, kemudian aktivasi zeolit alam Bayah, dan proses adsorpsi minyak jelantah dengan massa zeolit 15 dan 30% b/b. Hasil penelitian menunjukkan karakteristik minyak jelantah sebelum diadsorbsi memiliki bilangan asam 1.51% dan bilangan peroksida 28.43 mek $O_2$/kg yang telah sesuai dengan parameter baku mutu minyak goreng. Pengaruh zeolit teraktivasi alkali (NaOH 15 dan 30 %) terhadap minyak jelantah menurunkan kadar asam lemak bebas menjadi 0.98% dan 0.75%, serta bilangan peroksida masing-masing adalah 19.54 mek $O_2$/kg dan 18.38 mek $O_2$/kg. Semakin besar jumlah zeolit yang ditambahkan, maka semakin rendah bilangan asam dan bilangan peroksida dalam minyak, sehingga meningkatkan kualitas minyak sebagai bahan baku alkyd resin.

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## 1. Introduction

Indonesian people's consumption of cooking oil has increased 29.77%, from 3.89 million tons in 2014 to 14.72 million tons in 2019 [1]. Waste cooking oil is kitchen waste that contains hazardous compounds, if it is disposed of without prior processing it will pollute the environment and cause water and soil pollution [2]. Several phenomena affect oil quality, including qualitative changes that can be evaluated by visual inspection. Color is a physical indicator for the food industry and the phenomenon of chemical change such as increases in the concentration of free fatty acids, polar materials, polymeric compounds, and a decrease in the composition of unsaturated fatty acids. It had been influenced by three types of chemical reactions: hydrolysis, oxidation, and polymerization. Hydrolysis involves the breakdown of triglycerides in the presence of water and steam. This process produces monoglycerides, diglycerides, free fatty acids, and glycerol. Hydrolysis depends on the temperature of the oil, the area of the interface between the oil phase and the solution phase, and the amount of water and steam. Free fatty acids and lower molecular weight acid products arising from fat oxidation enhance the hydrolysis process in the presence of steam during frying. Hydrolysis can be determined by the saponification number of the oil [3].

The characteristics of waste cooking oil are not good for reuse in any process. The waste cooking oil needs pretreatment to remove or separate harmful compounds which interfere with the process of forming products from the waste cooking oil. Therefore, the partial purification of cooking oil before further application can be carried out by various physical and chemical methods such as filtration, adsorption, chromatography, and extraction by solvents [4]. The process of adsorption of a liquid, or gas which bound to a solid or liquid (adsorbent, adsorbent) and finally forms a thin layer or film (adsorbent substance, adsorbate) on its surface, this process is called adsorption. Adsorption is classified into chemical adsorption and physical adsorption. Physical adsorption occurs due to the "Van der Walls" force and is reversible. The adsorbent used in physical adsorption must have a large surface area for the solute to collect. While the chemical adsorption is few irreversible. The distance of the molecules in a solid is the same in each direction, so the attractive forces between one molecule and the other around it are balanced [5].

The potential of Banten's natural resources in the form of zeolite minerals originating from Bayah has a megascopic appearance of brownish-white, greenish-white, dark green, light gray, dark gray, and brownish after weathering. Bayah is a sub-district in the district of Lebak, Banten. Zeolite is a natural material in the form of hydrated alumina silicate crystals whose composition contains alkaline or alkaline earth cations in the form of a three-dimensional framework. Zeolites have acidic properties and have molecular-sized pores. Zeolites contain three components, namely exchangeable cations, an alumina silicate framework, and water. Bayah natural zeolite can be used in the adsorption process of used cooking oil to reduce the value of free fatty acids and peroxide value in used cooking oil. This adsorbent has advantages, namely cheap, easy to obtain, and can increase the utilization of regional resource potential. Bayah natural zeolite is a non-metallic mineral commodity or multi-use industrial mineral because it has physical and chemical properties as an absorber, ion exchanger, molecular filter, and catalyst [6].

The interest in converting waste cooking oil (WCO) into raw materials has grown exponentially. This idea is driven by the increasing number of WCO applications in many countries and regulations regarding waste management. From an industrial perspective, WCO's simple chemical composition makes it suitable for forming valuable chemicals in the production of additives in the paints, coatings, plastics, fuels, materials, bio-lubricant, resins, and biodegradable polymer industries. The development of new recycling processes, as well as process optimization, represent a priority for applied chemistry, chemical engineering, and materials science [7]. This paper studied the effect of the mass of Bayah natural zeolite (w/w %) on the characteristics of waste cooking. The Bayah natural zeolite as adsorbent reduces the value of free fatty acids and peroxide numbers.

Used cooking oil derived from vegetable oil waste can be used as raw material for the synthesis of Alkyd resins. Alkyd resin is additive for binder the paint, coating, plastics, adhesives, and other industries. Alkyd resins are widely used in the anti-corrosion coating industry.

## 2. Research Method

### 2.1. *Preparation of Waste Cooking Oil and Bayah Natural Zeolite*

Waste cooking oil was obtained from street vendors in Cilegon, Banten. The waste cooking oil collected was mixed in a container, then the waste cooking oil was filtered with filter paper and then analyzed for the chemical content of the ingredients, namely acid number, free fatty acid, and peroxide number, and characterized by FT-IR. The type of FTIR is Prestige-21 Shimadzu. FTIR was performed using the length medium wave (Mid-IR) that is 4000 - 400 $cm^{-1}$, analysis performed to investigate catalyst morphology and composition with SEM-EDX (JEOL 6360 LA). For zeolite preparation, Bayah Natural Zeolite (BNZ) was obtained from Bayah, Banten. The BNZ was activated is 500 grams and used for absorbent about 15 and 30% w/w from waste cooking oil. BNZ was crushed, then screened to get smaller sizes, then sieved to 80 mesh.

### 2.2. *Bayah Natural Zeolite Activation*

BNZ adsorbents were refluxed with 750 mL of 0.75 N NaOH solution at a temperature of $90^oC$ for 2 hours, then washed with hot water until the pH was neutral, then dried in an oven at $110^oC$ for 1 hour, then calcined at $400^oC$ for 2 hours.

### 2.3. *Cooking Oil Adsorption*

The waste cooking oil was filtered with filter paper, then the BNZ adsorbent was added to the oil with a percentage of 0, 15, and 30% w/w, then heated at $80^oC$ for 1 hour, after it was filtered with filter paper.

## 3. Result and Analysis

### 3.1. *Characteristics of Cooking Oil*

Waste cooking oil is obtained from street vendors and household waste. The waste cooking oil that has been collected from traders and household waste is analyzed physically and chemically. The following characteristics of waste cooking oil is shown in Table 1.

**Table 1.** Characteristics of cooking oil.

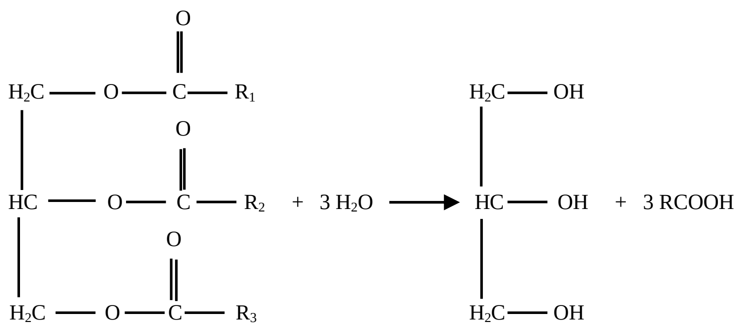
| Parameter | Waste cooking oil |
|---|---|
| Odor | Rancid |
| Color | Yellowish-brown |
| Density | 0.8976 g/ml |
| Viscosity | 3910 cp |

| Parameter | Waste cooking oil |
|---|---|
| Free fatty acid | 1.46% |
| Peroxide number | 28.43 mek $O_2$/kg |

The physical quality of the oil can be seen from the color and smell. The filtered cooking oil is brown-yellow and has a rancid smell, while the cooking oil that is still suitable for use is golden yellow and smells normal (not rancid). This physical change occurs because of the oxidation reaction. Oxidation reactions occur between oxygen and the double bonds of triglycerides or oils. Cooking oil is usually added with antioxidants which function to inhibit the oxidation reaction between oxygen and triglycerides in the oil. The brown color of waste cooking oil can be caused by the bond between carbohydrate and protein molecules. The occurrence of this bond is also known as the Maillard reaction, which is a reaction between the carbonyl group and the amino group of the protein. Heating too high and repeated causes the polymerization reaction and the Maillard reaction which causes the oil to thicken and darken in color. This is inline with the research that has been carried out in [8].

Based on the analysis of waste cooking oil shown in Table 1, It was contains the values of free fatty acids and peroxide numbers. It show the quality of the oil for determine the chemical events in the oil. If oil contains a free fatty acid value and high peroxide number must be recycled before reuse. This study, the acid value-free fat in this study was 1.46%, it inline in [9], that waste cooking oil has a free fatty acid value greater than 0.3%.

The hydrolysis process of oil will be formed fee fatty acid. The mechanism reaction is shows in Figure 1. The triglycerides are hydrolyzed to glycerol and free fatty acids in the presence of water.




Tryglycerid Water Glycerol Free fatty acid

**Figure 1. Reaction process alcoholysis formation of monoglycerides.**

The value of free fatty acids in waste cooking oil was determined by titration KOH or NaOH. The ethanol was added for dissolve fat or oil in the sample, so it can react with alkaline bases. The concentration of ethanol is 95-96%. The method of determining free fatty acids using direct acid-base titration with phenolphthalein (PP) indicator. The reaction was showed as follow.

$$R\text{-}COOH + NaOH \rightarrow R\text{-}COONa + H_2O$$

Another parameter besides free fatty acids is the number of peroxides. Unsaturated fatty acids can bind oxygen in the double bond to form peroxides and then aldehyde compounds, lactone compounds, and acrolein compounds are formed. The peroxide number was show the oxidation of unsaturated fatty acids in cooking oil by oxygen and produces a peroxide compound. This study the value of peroxide is 28.43 mek $O_2$/kg, this result is greater than [10]. The method used to determine the content of peroxide compounds is iodometric titration. Oil is dissolved by a non-polar solvent, in a solution that has an acidic pH, alkaline iodide will react completely. Iodometric titration occurs KI oxidation reaction by peroxide compounds to produce $I_2$. Titration was carried out using a starch indicator.

$$R\text{-}OOH + 2\ KI + H_2O \rightarrow R\text{-}OH + I_2 + 2\ KOH$$

The amount of $I_2$ released is equivalent to the number of peroxide compounds, then this $I_2$ compound will be titrated by sodium thiosulfate which is a reducing agent which will reduce $I_2$ to $I^-$. This iodometric titration is a type of indirect titration [10].

$$I_2 + 2\ Na_2S_2O_3 \rightarrow 2\ NaI + Na_2S_4O_6$$

The oxidation of oil produces oxygen-derived form free radicals and hydroxylation products which in turn will become FFA, aldehydes, and ketones that induce organ toxicity. Analyzing of functional groups of compounds in a solution determined with FT-IR by absorption of molecular vibrations in compounds at certain wavelengths. The infrared absorption intensity spectrum of the starting raw material waste cooking oil is presented on Figure 2

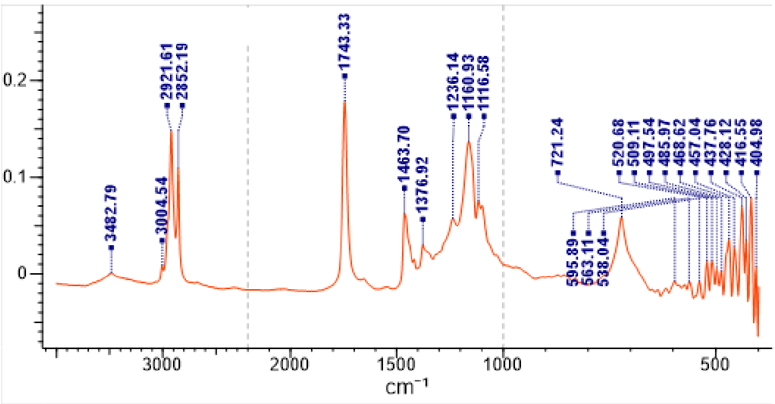



**Figure 2. Graph of wavelength and peak of vibration absorption function group cooking oil without pretreatment.**

The results of the graph in Figure 2 are compared with the oil literature from analyzing of FTIR and than the data is presented in Table 2.

**Table 2.** The wavelength of vibration absorption function group of cooking oil without pretreatment.

| | Oil compound wavelength ($cm^{-1}$) | |
|---|---|---|
| **Functional group** | **Research result** | **Literature [11]** |
| O-H | 3482.79 | 3471 |
| C-H unsaturated | 3004.54 | 3007 |
| C-H saturated | 2921.45 | 2924 |
| C=O | 1743.843 | 1745 |
| C-O-C triglyceride | 1161.019 | 1163 |

The results shows that vibrational peak of the O-H alcohol functional group is 3482.79 $cm^{-1}$ which came from the detection of the O-H functional group in the glycerol compound which is the result of the hydrolysis of triglycerides in oil. The unsaturated C-H functional group is 3004.54 $cm^{-1}$ and the saturated C-H functional group is 2921.453 $cm^{-1}$ came from the detection of saturated and unsaturated fatty acids in triglycerides. The C=O functional group at 1743.843 $cm^{-1}$ and the C-O-C ester group at 1161.019 came from the carbonyl and ester functional groups in triglycerides. These results are inline in [11].

### 3.2. *Characteristics of Bayah Natural Zeolite activated*

Waste cooking oil contains of glycerol and free fatty acids (FFA). The FFA was formed from the heating of oil which causes the hydrolysis process. The FFA reacts with glycerol and form monoglycerides. FFA is carboxylic compounds a have a polar side, namely a carboxyl group (-COOH). In synthesis alkyd resin, the glycerol reacted with triglycerides to produce monoglycerides. Therefore, the FFA from the hydrolysis product needs to be reduced first because it can inhibit the polyesterification reaction.

The treatment for reduction of FFA is adsorption process with several types of adsorbents, such as zeolite. The Natural zeolite is a natural porous mineral that has a large surface area and high selectivity. However, natural zeolite has a weakness, such as the adsorption capacity of natural zeolite is low due to impurities and the presence of an irregular alumina-silicate framework structure. Organic, inorganic, and water molecule impurities present in natural zeolite was blocked the active site on the pore surface so the adsorbate molecule cannot be absorbed, and the zeolite needs to be activated to increase the value of cation exchangeability.

The activated zeolite can be characterized by observing the important functional groups that play a role in the absorption of FFA. In this study, a graph of the vibrational spectrum of the functional group was obtained which is shown in Figure 3.

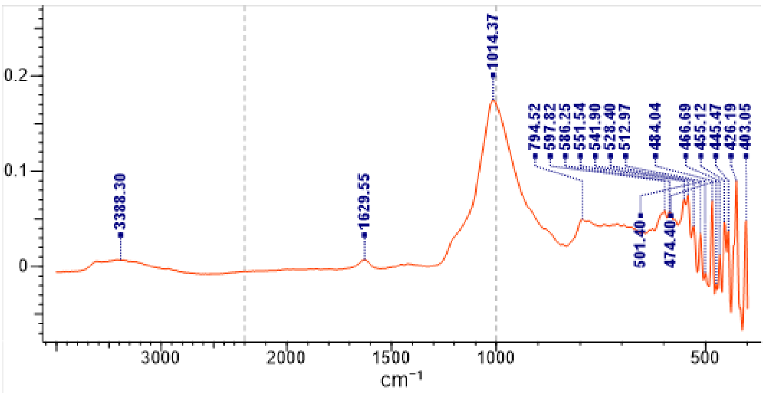



**Figure 3. Graph of wavelength and peak of vibration absorption of functional groups in NaOH activated zeolite 0.75 N.**

The wavelength data of the vibrational absorption of molecular functional groups is show in Table 3.

**Table 3.** Functional groups on NaOH-activated Zeolite 0.75 N.

| | Compound wavelength in zeolite ($cm^{-1}$) | |
|---|---|---|
| **Functional groups** | **Research result** | **Literature [12]** |
| Si-O-Al | 464.758 | 462.92 |
| Si-O | 794.966 | 794.67 |
| Si-O-Si | 1014.370 | 1049.28 |
| $TiO_4$ | 1165.752 | 1211.29 |

Based on Table 3, the results shows the vibrations of the Si-O-Al functional group is a wavelength of 464.758 $cm^{-1}$, the Si-O functional group at a wavelength is 794.966 $cm^{-1}$, the Si-O-Si functional group at a wavelength is 1014.370 $cm^{-1}$, and the $TiO_4$ functional group at a wavelength is 1165.752 $cm^{-1}$. These results inline in [12]. The morphology of BNZ particles by chemical activation using 0.75 N NaOH and physical activation by heating at a temperature of 400°C was determined with Scanning Electron Microscopy (SEM). The result as presented in Figure 4.

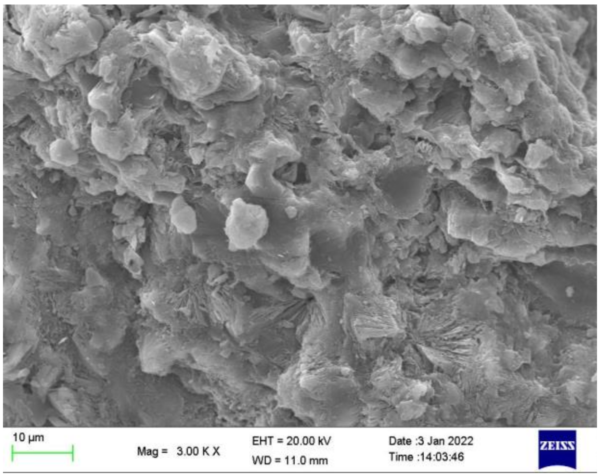



**Figure 4. Scanning electron microscopy analysis results.**

Based on Figure 4 that the results analysis SEM with 5000 times magnification shows the surface of the BNZ activated with a base looks hollow. This is due to the loss of metal ions covering the BNZ surface. The zeolite content quantitatively from each material preparation was evaluated by EDX. The quantitative analysis of the BNZ element content and Si/Al ratio is presented in Table 4 – 5, respectively.

**Table 4.** Elemental content in NaOH activated zeolite 0.75 N.

| **Component** | **Content (wt.%)** |
|---|---|
| Carbon | 1.29 |
| Oxigen | 52.07 |
| Natrium | 1.98 |
| Magnesium | 0.60 |
| Aluminum | 7.05 |
| Silicon | 31.74 |
| Kalium | 2.44 |
| Calcium | 1.97 |
| Iron | 0.85 |

**Table 5.** The ratio of Si/Al Zeolite.

| **Component** | **Activation of NaOH 0.75 N research results (wt.%)** | **Activation $H_2SO_4$ 1 M [13]** |
|---|---|---|
| Si/Al | 4.490 | 6.22% |

Bayah natural zeolite which was activated with NaOH the Si/Al ratio is smaller than activation with HCl. Natural zeolite which is activated with base can cause desilication so the Si/Al ratio will decrease, and the zeolite becomes more hydrophilic. This is inline in [12] and [14], which states that the Si/Al ratio in base-activated zeolite is lower than the ratio of Si/Al which is activated by acid and without activation. This property can be used for the adsorption process of hydrophilic compounds, one of which is fatty acids.

The chemical activation process with NaOH will dissolving of silica which is one of the components in the zeolite framework. The dissolution of silica will cause changes in the structure of the zeolite and reduce silica in the zeolite framework so that the Si/Al ratio decreases. In addition to decreasing the

Si/Al ratio, activation with NaOH also aims to remove certain ions from the zeolite framework and replace them with Natrium ions so that the zeolite has a condition that is closer to the homoiconic form, this is inline in [12]. While the physical activation aims to remove organic impurities, enlarge pores, and expand the surface. Physical activation was carried out by heating Bayah natural zeolite. The heating to evaporate the water trapped in the crystal pores so that the number of pores and the specific surface area increases, this statement is in line on [14].

### 3.3. *Oil Processing with Zeolite and Characterization of Pretreated Oil*

The results of the characterization of raw material used cooking oil after being adsorbed with Bayah natural zeolite using various mass ratios are shown in Table 6.

**Table 6.** Comparison of raw material cooking oil with pretreated cooking oil.

| Parameter | N/A ZAB | Adsorption 15% ZAB | Adsorption 30% ZAB |
|---|---|---|---|
| Odor | Not Normal | Not Normal | Not Normal |
| Color | Yellowish-brown | Yellow-brown | Yellow-brown |
| Free fatty acid | 1.46 % | 0.96 % | 0.74 % |
| Peroxide Number | 28.43 mek $O_2$/kg | 19.54 mek $O_2$/kg | 18.38 mek $O_2$/kg |
| Acid Number | 3.34 mg KOH/gr | 2.07 mg KOH/gr | 1.62 mg KOH/gr |
| Density | 0.8976 g/ml | 0.8984 g/ml | 0.8988 g/ml |
| Viscosity | 3910 cp | 3909 cp | 3909 cp |

Based on Table 6, the acid number in the oil changed from 3.34 mg KOH/gr to 2.07 mg KOH/gr and 1.62 mg KOH/gram after being adsorbed using 15% w/w zeolite and 30% w/w zeolite, respectively. Based on the results, it can be seen that the amount of zeolite used is directly proportional to the percentage decrease, i.e. 38% and 51.49% for the addition of 15% w/w and 30% w/w zeolite, respectively. This result is greater than the study in [16] which used zeolite without an activation process which resulted in a decrease in FFA of 0.003%. In addition, these results are following the theory that the greater the amount of adsorbent, the greater the surface area of the adsorbent so the greater the free fatty acids absorbed.

The content of free fatty acids decreased with an increase in the amount of zeolite used. This is to the adsorption theory that the factors that affect the adsorption process include the size of the adsorbent and the number of adsorbents that affect the surface area, so that the smaller and the larger the number of adsorbents, the surface area will increase, this is by the literature research in [17]. The increase in the surface area of the zeolite resulted in more FFA being absorbed so that the remaining FFA in the oil became less. The low number of free fatty acids in the oil indicates the better the quality of the oil.

The peroxide value changed from the original 28.43 mek $O_2$/kg to 19.54 mek $O_2$/kg after being adsorbed with 15% mass zeolite, while 18.38 mek $O_2$/kg after being adsorbed using 30% mass zeolite. The low number of peroxides indicates that the fewer peroxide compounds in the oil, so the quality of the oil is getting better because the lower the peroxide compounds that cause oil damage.

## 4. Conclusion

The characteristics of waste cooking oil before pretreatment has an acid and peroxide value is 1.46% and 28.43 mek $O_2$/kg, respectively. The free fatty acid value and peroxide values are 0.6% and 10 mek $O_2$/kg. The Bayah natural zeolite used for absorption of the impurities of waste cooking oil. It was activated with 0.75 N NaOH is hydrophilic to free fatty acids in the oil, and activated zeolite has a Si/Al ratio is 4.490% wt. The effect of zeolite activated with alkaline 15 % w/w and 30% w/w caused the free fatty acid content decreases to 0.96% and 0.74%, and the peroxide value decreases to 19.54 mek $O_2$/kg and 18.38 mek $O_2$/kg. The amount of zeolite added is not proportional to the acid number and peroxide value. The quality of the oil was a good performance as the feedstock of alkyd resin.

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