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Preparation and Characterization of Natural Zeolite Minerals to Reduce Free Fatty Acid Levels in Used Cooking Oil

(Persiapan dan Karakterisasi Bahan Tambang Zeolit Alam untuk Menurunkan Kadar Asam Lemak Bebas pada Minyak Jelantah)

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Abstract

Zeolite as a catalyst can increase its activity by carrying out physical and chemical activation processes. Physical activation can be activated by reducing the size of the zeolite through grinding and sieving methods, from the physical activation process it can increase the surface area of the zeolite. In this study, zeolite was chemically activated by acid activation method by immersing natural zeolite with HCl and HF. The oxide compound in the activated zeolite was characterized using XRF. The activated zeolite was used to reduce the free fatty acid levels with variations in the addition of 7 to 10 grams of zeolite. The content of free fatty acids is then compared with the quality requirements of cooking oil according to SNI 01-3741-2013. The results of the research conducted showed that there were differences in the content of oxide compounds in zeolite before and after activation with differences in SiO₂ content, namely 69.4% before activation and an increase of 82.1% after activation. The results of the state test including physical properties, odor, and taste tests of used cooking oil that have been purified indicate that used cooking oil that has been purified meets the quality requirements of cooking oil based on SNI 01-3741-2013. Used cooking oil that has been purified has a normal taste, does not smell, rancid and yellow. The content of free fatty acids meets the requirements of SNI 01-3741-2013 for all variations of the addition of zeolite with the highest decreasing value obtained at the addition of 10 g of zeolite with a free fatty acid content value of 0.22%.

Keywords: Acid Activation; Zeolite, Used Cooking Oil; Free Fatty Acid Levels

Sari

Zeolit sebagai katalis dapat meningkatkan aktivitasnya dengan melakukan proses aktivasi fisik dan kimia. Aktivasi fisik dapat diaktifkan dengan cara memperkecil ukuran zeolit melalui metode penggilingan dan pengayakan, dari proses aktivasi fisik dapat meningkatkan luas permukaan zeolit. Pada penelitian ini zeolit diaktivasi secara kimia dengan metode aktivasi asam dengan merendam zeolit alam dengan HCl dan HF. Senyawa oksida pada zeolit teraktivasi dikarakterisasi menggunakan XRF. Zeolit yang telah diaktivasi digunakan untuk menurunkan kadar asam lemak bebas dengan variasi penambahan zeolit 7 sampai 10 gram. Kandungan asam lemak bebas kemudian dibandingkan dengan syarat mutu minyak goreng menurut SNI 01-3741-2013. Hasil penelitian yang dilakukan menunjukkan bahwa terdapat perbedaan kandungan senyawa oksida pada zeolit sebelum dan setelah aktivasi dengan perbedaan kandungan SiO₂ yaitu 69,4% sebelum aktivasi dan meningkat 82,1% setelah aktivasi. Hasil uji keadaan meliputi uji sifat fisik, bau, dan rasa minyak goreng bekas yang dimurnikan menunjukkan bahwa minyak goreng bekas yang telah dimurnikan memenuhi syarat mutu minyak goreng berdasarkan SNI 01-3741-2013. Minyak goreng bekas yang telah dimurnikan memiliki rasa yang normal, tidak berbau, tengik dan kuning. Kandungan asam lemak bebas memenuhi syarat SNI 01-3741-2013 untuk semua variasi penambahan zeolit dengan nilai penurunan tertinggi diperoleh pada penambahan 10 g zeolit dengan nilai kadar asam lemak bebas sebesar 0,22%.

Kata-kata kunci: Aktivasi Asam; Zeolit; Minyak Jelantah; Kadar Asam Lemak Bebas

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I. INTRODUCTION

Zeolites are a group of minerals which are defined as one of the 50 types of non-metallic minerals or industrial minerals. Until now, more than 50 natural zeolite-forming minerals, the types of zeolite minerals found in Indonesia are modernite and klipno-ptilolite. This natural zeolite is formed from the reaction between fine-grained and rhyolitic acid tuff rocks with pore water or meteoric water (rainwater). This mineral is a hydrated aluminosilicate group with the main elements consisting of cations, alkalis, and alkaline

earths, having pores that can be filled by molecular. The air content trapped in the zeolite cavity usually ranges from 10-50%. When hydrated, the cations in the cavity will be enveloped in water molecules, these water molecules are unstable or easily released [1, 2].

Natural zeolite has quite a lot of pores, which is about 30% more than its volume and is mixed with impurities other than zeolite, therefore natural zeolite needs to be activated and modified to increase its activity. Through the activation process, impurities can be removed and change the Si/Al ratio. The

activation process can be done by heating and adding acid or base [3, 4]. Physical activation is carried out by reducing grain size, sieving, and heating at high temperatures which aims to remove organic impurities, increase pore size and expand the surface [5, 6]. The physical activation process can be carried out by calcining natural zeolite at a temperature of 600°C. Chemical activation can be carried out using a solution of hydrochloric acid (HCl) or sulfuric acid (H₂SO₄), as well as a solution of sodium hydroxide (NaOH) which aims to clean the surface of the pores, removing impurities. The addition of acid will increase the porosity so that the zeolite activity increases [7].

The ability of zeolite to absorb molecules depends on the nature of the molecule, the ability of the zeolite to exchange ions, the acidity of the zeolite surface, and the water content in it. All of these factors are determined by the condition of the pores in the zeolite, which in turn can position the zeolite as a catalyst in certain reactions. The tetrahedral zeolite framework is usually partially negatively charged, due to the influence of the isomorphic change from Si⁴⁺ to Al³⁺. The tetrahedral skeleton is then neutralized by cations that are exchangeable, resulting in ionic bonds between the tetrahedral skeletons. To increase the ability of zeolite as an absorbent agent, activation is carried out both physically and chemically [8,9,10].

Zeolite is reacted with acid compounds through an immersion process for a while until the impurities are gone and rinsed until neutral test results are obtained, after the zeolite is neutral, then the activated zeolite is dried [11,12]. The empty space in the zeolite cavity causes zeolite to be used as a catalyst [13,14]. Activating zeolite by adding NaOH and using it as an adsorbent in the purification of used cooking oil. The results showed that with the addition of zeolite, the impurities in the used cooking oil could be removed, marked by a decrease in free fatty acid levels [15].

Used cooking oil is an oil that has been used repeatedly in the food processing process. The result of repeated processing causes the quality of used cooking oil to decrease. The quality of used cooking oil can be seen from the formation of saturated fatty acids which is characterized by the number of double bonds formed in the structure of used cooking oil. The double bonds formed will cause the used cooking oil to easily oxidize and make it more difficult to bond with other materials. Cooking oil that has been oxidized will produce carcinogenic compounds such as free fatty acids which if consumed will be harmful to health [16].

In this research, activated zeolite by immersing zeolite with acid and characterization with XRF tests. Furthermore, the activated zeolite was used as an adsorbent in the used cooking oil refining process and tested for quality improvement by determining the value of free fatty acid content.

II. METHOD

The research was conducted at the Chemistry Laboratory Trisakti University, Jakarta. Zeolite activation is divided into two stages, preparation and characterization. Washing, filtering, drying, and calcining were carried out to prepare the zeolite. Characterization using XRF to see the difference in the content of zeolite oxide compounds before and after being oxidized. The activated zeolite was then used as an adsorbent in the used cooking oil purification process.

2.1 Materials and Equipment

The materials used in this study were Sukabumi natural zeolite, HF (Merck), HCl (Merck), cooking oil, glacial acetic acid (Merck), chloroform (Merck), KI (Merck), starch, Na₂S₂O₃ (Merck). Merck, alcohol (Merck), Phenolphthalein indicator (Merck), NaOH (Merck) KOH (Merck), and aquadest.

XRF was used in this research for the characterization of natural zeolite before and after activation. A series of reflux apparatus and glassware were used in the preparation of zeolite and quality analysis of the purified used cooking oil, and the oven was used in the water content test of used cooking oil.

2.1.1 Acidic Activation of Zeolite

Zeolite was ground and sieved to a size of 100 mesh. Then the zeolite was washed with distilled water and soaked using hydrofluoric acid. Zeolite was soaked with acid and then rinsed with distilled water until the pH is neutral. Zeolite with neutral pH was then refluxed with HCl and stirred continuously. The zeolite was then filtered and rinsed with distilled water until the pH was neutral and dried at 130°C for 3 hours.

2.1.2. Purification of Used Cooking Oil

A total of 100 g of used cooking oil was put into a beaker and zeolite was added with variations in the addition of 7, 8, 9, and 10 g. The mixture of used cooking oil with zeolite was then stirred using a hotplate stirrer with a stirring speed of 250 rpm and a temperature of 100-110°C for 60 minutes. The homogenized used cooking oil is then filtered using Whatman 42 filter paper. The results of the filtering of used cooking oil are then tested for quality by performing appearance tests (color, odor, and taste) in the form of physical properties test that can be observed from the purified used cooking oil in the form of color, smell, and taste and free fatty acid content test compared with SNI 01-3741-2013 [17].

2.1.3. Determination of Free Fatty Acid Levels

The used cooking oil is stirred evenly and cultivated in a liquid state for easy extraction. The sample was weighed as much as 28 g and put into a 250 mL Erlenmeyer flask. 50 mL of hot neutral alcohol and 2 mL of phenolphthalein (PP) indicator

were added to the sample and immediately titrated using 0.100 N NaOH until a color change from colorless to pink did not disappear for 30 seconds.

III. RESULTS AND DISCUSSION

3.1 Zeolite Activation

Zeolite activation is carried out in two stages, namely physical and chemical activation. Physical activation by reducing the size of the zeolite through grinding and sifting processes. Zeolite was ground and sieved to obtain zeolite with a size of 100 mesh.

The function of reducing the size of the zeolite is to increase the surface area so that the increase in surface area will increase the catalytic activity of the zeolite because more reactants can be absorbed into the pores.

Chemical activation was carried out by soaking zeolite with acid. The acidity function is to clean the pore surface and remove impurity compounds [18]. The results of the activation that have been carried out were observed by comparing the chemical elements contained in the zeolite before and after activation as shown in Table 1 and Figure 1.

Table 1. The Content of Zeolite Oxide Compounds before and after Activation

No	Component	Zeolite oxide compounds before activation (%)	Zeolite oxide compounds after activation (%)
1	Al ₂ O ₃	13	2
2	SiO ₂	71.4	84.1
3	K ₂ O	8.22	9.21
4	CaO	7.61	6.73
5	TiO ₂	0.20	0.46
6	V ₂ O ₅	0.00	0.007
7	Cr ₂ O ₃	0.023	0.035

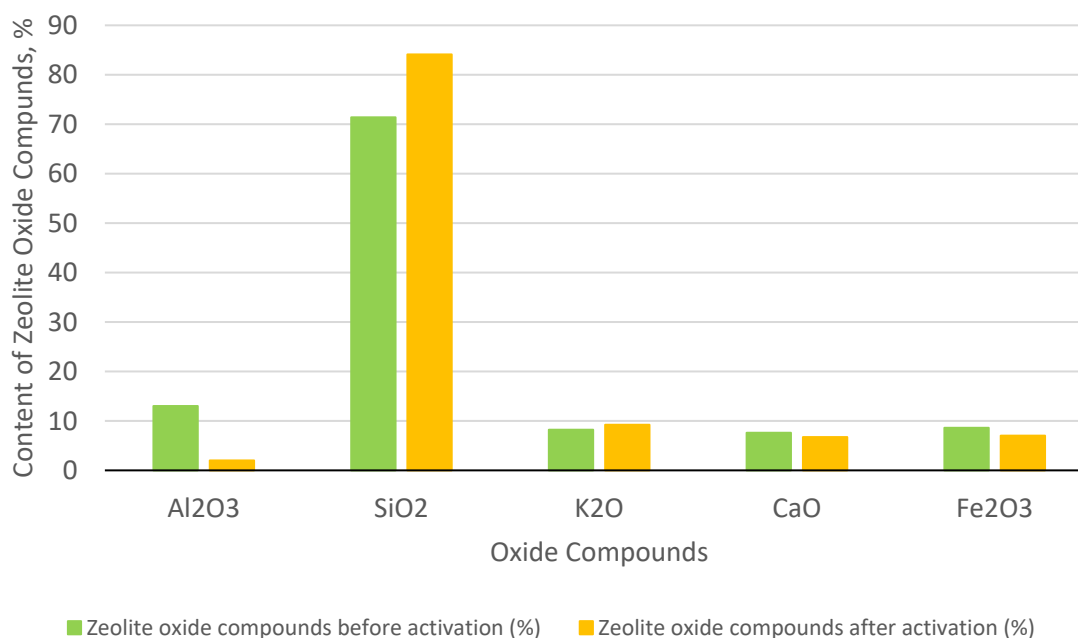


Figure 1. Zeolite compounds before and after activation

There are five main oxide compounds, namely oxide compounds with a higher percentage compared to other oxide compounds with a dominant percentage, namely Al₂O₃, SiO₂, K₂O, CaO, and Fe₂O₃. Silicon dioxide, which is the main compound in zeolite, experienced a significant increase in levels after activation, which increased from 69.4% levels before activation and increased to 82.1% after activation. This is because the impurities have

dissolved after activation with acid which causes the main content of the zeolite to increase.

3.2 Purification of Used Cooking Oil with Activated Zeolite

Zeolite has a crystalline structure that is hollow and interconnected with a large surface area which causes zeolite to be used as an adsorbent agent. The catalytic activity of zeolite as an adsorbent is

increasing with the activation of the zeolite both physically and chemically. The activated zeolite is then added to the used cooking oil to absorb impurities that have been adsorbed into the oil that has been used to fry food repeatedly.

In the purification of used cooking oil with zeolite, stirring is carried out on the sample which serves to

homogenize the mixture that has been formed. The application of stirring at a constant speed resulted in more and more impurities from the used cooking oil being absorbed by the zeolite. This can be seen through the visible physical properties of used cooking oil before and after being purified, including color, odor, and taste are shown in Figure 2.



Figure 2. Used cooking oil before being purified (a) and used cooking oil after being purified (b)

The state test of used cooking oil was carried out by comparing the physical properties of used cooking oil before and after purification by observing the physical properties of used cooking oil in the form of color, smell, and taste. The results of the observation of the condition of used cooking oil were then compared with the quality requirements of cooking oil based on SNI 01-3741-2013. From the results of the comparison of the state of the purified used cooking oil with the SNI standard, it was found that based on the physical properties observed the used cooking oil complied with SNI 01-3741-2013, namely the smell of the used cooking oil that was purified normal and not rancid, the taste of the used cooking oil that was purified was normal, and the properties of the used cooking oil. The last physical observation observed was the color of the purified used cooking oil which showed a pale yellow color.

3.3 Quality Test of Refined Cooking Oil

The quality of the purified used cooking oil was tested by measuring the peroxide number, free fatty

acid content, acid number, and water content and compared with the quality requirements of cooking oil based on SNI 01-3741-2013. The quality of used cooking oil that was purified with zeolite was compared with the quality of used cooking oil without purification.

3.4 Free Fatty Acid Level

One of the parameters that indicate the declining quality of the oil is the high number of free fatty acids caused by repeated heating. Repeated heating of cooking oil will cause hydrolysis and oxidation of cooking oil which causes the formation of glycerol and free fatty acids. The high content of fatty acids causes an unpleasant taste in cooking oil. In addition, the high content of free fatty acids will cause disease, one of which is increased cholesterol levels in the blood. The results of the measurement of free fatty acid levels in used cooking oil samples before and after purification are shown in Table 2.

Table 2. Free Fatty Acid Test Results

Sample	Variation of addition of zeolite (gr)	Free fatty acid (%)	SNI 01-3741-2013 (%)
Initial sample	0	0.545	0.3
Sample 1	7	0.23	
Sample 2	8	0.23	
Sample 3	9	0.227	
Sample 4	10	0.22	

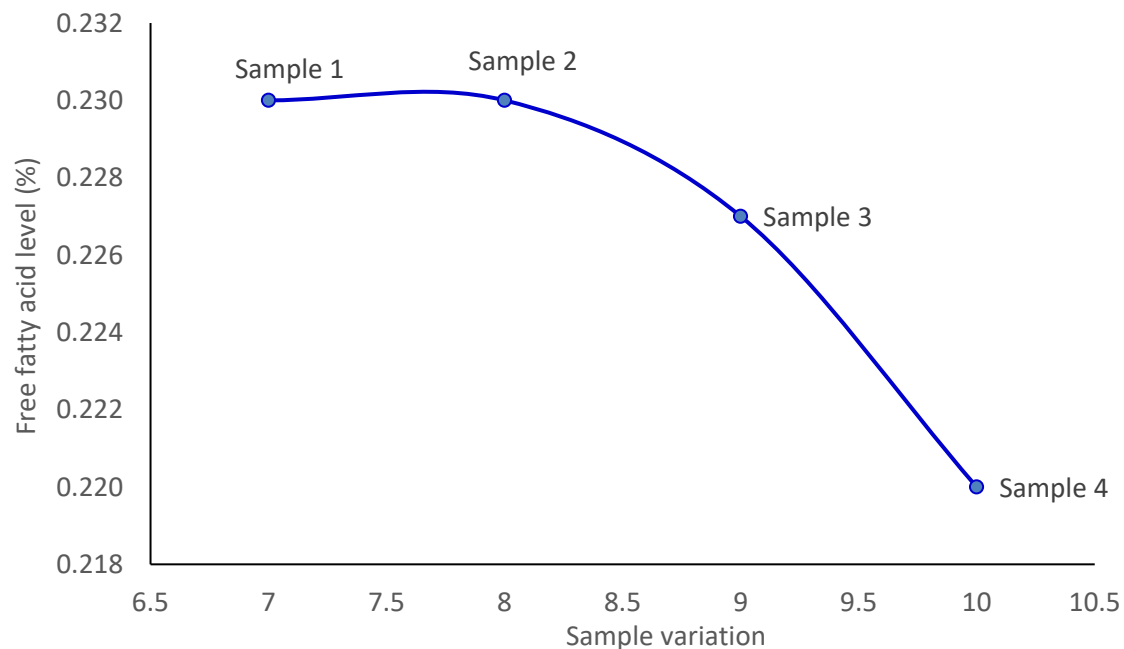


Figure 3. The relationship between variations in the addition of zeolite (gr) with free fatty acid levels (%)

From Table 2, it is known that the fatty acid content of used cooking oil before being purified does not meet the SNI standard for cooking oil quality, while the used cooking oil after being purified with zeolite meets the SNI standard, which is a maximum of 0.3% for all variations of addition with the largest decrease in free fatty acid levels with the addition of zeolite of 10 grams with a free fatty acid content of 0.22%. After the addition of zeolite, the free fatty acid content decreased and decreased with the increasing amount of zeolite added. The more zeolite added, the larger the surface area of the adsorbent, so the better the absorption rate. This is shown in Figure 3.

The same thing was also obtained by [19, 20, 21] who carried out the purification of used cooking oil with the adsorption process using Moringa seeds as an adsorbent. The results showed that the greater the number of Moringa seeds added, the smaller the measured free fatty acid levels.

IV. CONCLUSION

The content of Si and K elements increased after activation of the zeolite, while the content of Al, Ca, and Fe elements decreased after activation. The content of the oxide compound that is the main constituent of zeolite, namely SiO_2 , has increased levels after activation, which is 69.4% before activation and increases to 82.1% after activation.

The results of the state test on used cooking oil that has been purified with the addition of zeolite in the form of testing for odor, taste, and color indicate that the purified used cooking oil meets the quality requirements of cooking oil based on SNI 01-3741-

2013, namely producing purified oil with a normal taste, no rancid and pale yellow in color. The content of free fatty acids meets the requirements of SNI 01-3741-2013 for all variations of the addition of zeolite with the highest decreasing value obtained at the addition of 10 g of zeolite with a free fatty acid content value of 0.22% .

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Preparation and Characterization of Natural Zeolite Minerals to Reduce Free Fatty Acid Levels in Used Cooking Oil

by Wiwik Dahani

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Preparation and Characterization of Natural Zeolite Minerals to Reduce Free Fatty Acid Levels in Used Cooking Oil

(Persiapan dan Karakterisasi Bahan Tambang Zeolit Alam untuk Menurunkan Kadar Asam Lemak Bebas pada Minyak Jelantah)

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Abstract

Zeolite as a catalyst can increase its activity by carrying out physical and chemical activation processes. Physical activation can be activated by reducing the size of the zeolite through grinding and sieving methods, from the physical activation process it can increase the surface area of the zeolite. In this study, zeolite was chemically activated by acid activation method by immersing natural zeolite with HCl and HF. The oxide compound in the activated zeolite was characterized using XRF. The activated zeolite was used to reduce the free fatty acid levels with variations in the addition of 7 to 10 grams of zeolite. The content of free fatty acids is then compared with the quality requirements of cooking oil according to SNI 01-3741-2013. The results of the research conducted showed that there were differences in the content of oxide compounds in zeolite before and after activation with differences in SiO₂ content, namely 69.4% before activation and an increase of 82.1% after activation. The results of the state test including physical properties, odor, and taste tests of used cooking oil that have been purified indicate that used cooking oil that has been purified meets the quality requirements of cooking oil based on SNI 01-3741-2013. Used cooking oil that has been purified has a normal taste, does not smell, rancid and yellow. The content of free fatty acids meets the requirements of SNI 01-3741-2013. All variations of the addition of zeolite with the highest decreasing value obtained at the addition of 10 g of zeolite with a free fatty acid content value of 0.22%.

Keywords: Acid Activation; Zeolite, Used Cooking Oil; Free Fatty Acid Levels

Sari

Zeolit sebagai katalis dapat meningkatkan aktivitasnya dengan melakukan proses aktivasi fisik dan kimia. Aktivasi fisik dapat dilakukan dengan cara memperkecil ukuran zeolit melalui metode penggilingan dan pengayakan, dari proses aktivasi fisik dapat meningkatkan luas permukaan zeolit. Pada penelitian ini zeolit diaktivasi secara kimia dengan metode aktivasi asam dengan merendam zeolit alam dengan HCl dan HF. Senyawa oksida zeolit teraktivasi dikarakterisasi menggunakan XRF. Zeolit yang telah diaktivasi digunakan untuk menurunkan kadar asam lemak bebas dengan variasi penambahan zeolit 7 sampai 10 gram. Kandungan asam lemak bebas kemudian dibandingkan dengan syarat mutu minyak goreng menurut SNI 01-3741-2013. Hasil penelitian yang dilakukan menunjukkan bahwa terdapat perbedaan kandungan senyawa oksida pada zeolit sebelum dan setelah aktivasi dengan perbedaan kandungan SiO₂ yaitu 69,4% sebelum aktivasi dan meningkat 82,1% setelah aktivasi. Hasil uji keadaan meliputi uji sifat fisik, bau, dan rasa minyak goreng bekas yang dimurnikan menunjukkan bahwa minyak goreng bekas yang telah dimurnikan memenuhi syarat mutu minyak goreng berdasarkan SNI 01-3741-2013. Minyak goreng bekas yang telah dimurnikan memiliki rasa yang normal, tidak berbau, tengik dan kuning. Kandungan asam lemak bebas memenuhi syarat SNI 01-3741-2013. Semua variasi penambahan zeolit dengan nilai penurunan tertinggi diperoleh pada penambahan 10 g zeolit dengan nilai kadar asam lemak bebas sebesar 0,22%.

Kata-kata kunci: Aktivasi Asam; Zeolit; Minyak Jelantah; Kadar Asam Lemak Bebas

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I. INTRODUCTION

Zeolites are a group of minerals which are defined as one of the 50 types of non-metallic minerals or industrial minerals. Until now, more than 50 natural zeolite-forming minerals, the types of zeolite minerals found in Indonesia are modernite and klipno-ptilolite. This natural zeolite is formed from the reaction between fine-grained and rhyolitic acid tuff rocks with pore water or meteoric water (rainwater). This mineral is a hydrated aluminosilicate group with the main elements consisting of cations, alkalis, and alkaline

earths, having pores that can be filled by molecular. The air content trapped in the zeolite cavity usually ranges from 10-50%. When hydrated, the cations in the cavity will be enveloped in water molecules, these water molecules are unstable or easily released [1, 2].

Natural zeolite has quite a lot of pores, which is about 30% more than its volume and is mixed with impurities other than zeolite, therefore natural zeolite needs to be activated and modified to increase its activity. Through the activation process, impurities can be removed and change the Si/Al ratio. The

activation process can be done by heating and adding acid or base [3, 4]. Physical activation is carried out by reducing grain size, sieving, and heating at high temperatures which aims to remove organic impurities, increase pore size and expand the surface [5, 6]. The physical activation process can be carried out by calcining natural zeolite at a temperature of 600°C. Chemical activation can be carried out using a solution of hydrochloric acid (HCl) or sulfuric acid (H₂SO₄) as well as a solution of sodium hydroxide (NaOH) which aims to clean the surface of the pores, removing impurities. The addition of acid will increase the porosity so that the zeolite activity increases [7].

The ability of zeolite to absorb molecules depends on the nature of the molecule, the ability of the zeolite to exchange ions, the acidity of the zeolite surface, and the water content in it. All of these factors are determined by the condition of the pores in the zeolite, which in turn can position the zeolite as a catalyst in certain reactions. The tetrahedral zeolite framework is usually partially negatively charged, due to the influence of the isomorphic change from Si⁴⁺ to Al³⁺. The tetrahedral skeleton is then neutralized by cations that are exchangeable, resulting in ionic bonds between the tetrahedral skeletons. To increase the ability of zeolite as an absorbent agent, activation is carried out both physically and chemically [8,9,10].

Zeolite is reacted with acid compounds through an immersion process for a while until the impurities are gone and rinsed until neutral test results are obtained, after the zeolite is neutral, then the activated zeolite is dried [11,12]. The empty space in the zeolite cavity causes zeolite to be used as a catalyst [13,14]. Activating zeolite by adding NaOH and using it as an adsorbent in the purification of used cooking oil. The results showed that with the addition of zeolite, the impurities in the used cooking oil could be removed, marked by a decrease in free fatty acid levels [15].

Used cooking oil is an oil that has been used repeatedly in the food processing process. The result of repeated processing causes the quality of used cooking oil to decrease. The quality of used cooking oil can be seen from the formation of saturated fatty acids which is characterized by the number of double bonds formed in the structure of used cooking oil. The double bonds formed will cause the used cooking oil to easily oxidize and make it more difficult to bond with other materials. Cooking oil that has been oxidized will produce carcinogenic compounds such as free fatty acids which if consumed will be harmful to health [16].

In this research, activated zeolite by immersing zeolite with acid and characterization with XRF test. Furthermore, the activated zeolite was used as an adsorbent in the used cooking oil refining process and tested for quality improvement by determining the value of free fatty acid content.

II. METHOD

The research was conducted at the Chemistry Laboratory Trisakti University, Jakarta. Zeolite activation is divided into two stages, preparation and characterization. Washing, filtering, drying, and calcining were carried out to prepare the zeolite. Characterization using XRF to see the difference in the content of zeolite oxide compounds before and after being oxidized. The activated zeolite was then used as an adsorbent in the used cooking oil purification process.

2.1 Materials and Equipment

The materials used in this study were Sukabumi natural zeolite, HF (Merck), HCl (Merck), cooking oil, glacial acetic acid (Merck), chloroform (Merck), KI (Merck), starch, Na₂S₂O₃ (Merck), Merck), alcohol (Merck), Phenolphthalein indicator (Merck), NaOH (Merck) KOH (Merck), and aquadest.

XRF was used in this research for the characterization of natural zeolite before and after activation. A series of reflux apparatus and glassware were used in the preparation of zeolite and quality analysis of the purified used cooking oil, and the oven was used in the water content test of used cooking oil.

2.1.1 Acidic Activation of Zeolite

Zeolite was ground and sieved to a size of 100 mesh. Then the zeolite was washed with distilled water and soaked using hydrofluoric acid. Zeolite was soaked with acid and then rinsed with distilled water until the pH is neutral. Zeolite with neutral pH was then refluxed with HCl and stirred continuously. The zeolite was then filtered and rinsed with distilled water until the pH was neutral and dried at 130°C for 3 hours.

2.1.2 Purification of Used Cooking Oil

A total of 100 g of used cooking oil was put into a beaker and zeolite was added with variations in the addition of 7, 8, 9, and 10 g. The mixture of used cooking oil with zeolite was then stirred using a hotplate stirrer with a stirring speed of 250 rpm and a temperature of 100-110°C for 60 minutes. The homogenized used cooking oil is then filtered using Whatman 42 filter paper. The results of the filtering of used cooking oil are then tested for quality by performing appearance tests (color, odor, and taste) in the form of physical properties test that can be observed from the purified used cooking oil in the form of color, smell, and taste and free fatty acid content test compared with SNI 01-3741-2013 [17].

2.1.3. Determination of Free Fatty Acid Levels

The used cooking oil is stirred evenly and cultivated in a liquid state for easy reaction. The sample was weighed as much as 28 g and put into a 250 mL Erlenmeyer flask. 50 mL of hot neutral alcohol and 2 mL of phenolphthalein (PP) indicator

were added to the sample and immediately titrated using 0.100 N NaOH until a color change from colorless to pink did not disappear for 30 seconds.

III. RESULTS AND DISCUSSION

3.1 Zeolite Activation

Zeolite activation is carried out in two stages, namely physical and chemical activation. Physical activation by reducing the size of the zeolite through grinding and sifting processes. Zeolite was ground and sieved to obtain zeolite with a size of 100 mesh.

function of reducing the size of the zeolite is to increase the surface area so that the increase in surface area will increase the catalytic activity of the zeolite because more reactants can be absorbed into the pores.

Chemical activation was carried out by soaking zeolite with acid. The acidity function is to clean the pore surface and remove impurity compounds [18]. The results of the activation that have been carried out were observed by comparing the chemical elements contained in the zeolite before and after activation as shown in Table 1 and Figure 1.

Table 1. The Content of Zeolite Oxide Compounds before and after Activation

No	Component	Zeolite oxide compounds before activation (%)	Zeolite oxide compounds after activation (%)
1	Al ₂ O ₃	13	2
2	SiO ₂	71.4	84.1
3	K ₂ O	8.22	9.21
4	CaO	7.61	6.73
5	TiO ₂	0.20	0.46
6	V ₂ O ₅	0.00	0.007
7	Cr ₂ O ₃	0.023	0.035

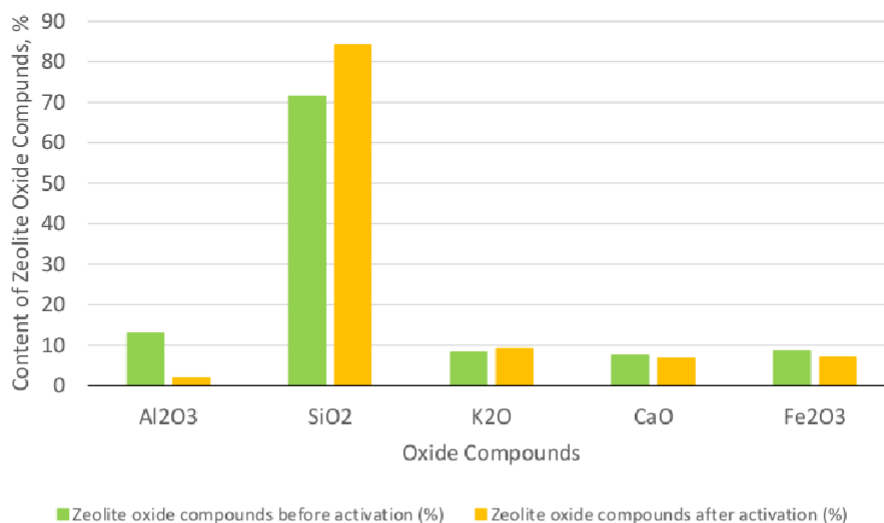


Figure 1. Zeolite compounds before and after activation

There are five main oxide compounds, namely oxide compounds with a higher percentage compared to other oxide compounds with a dominant percentage, namely Al₂O₃, SiO₂, K₂O, CaO, and Fe₂O₃. Silicon dioxide, which is the main compound in zeolite, experienced a significant increase in levels after activation, which increased from 69.4% levels before activation and increased to 82.1% after activation. This is because the impurities have

dissolved after activation with acid which causes the main content of the zeolite to increase.

3.2 Purification of Used Cooking Oil with Activated Zeolite

Zeolite has a crystalline structure that is hollow and interconnected with a large surface area which causes zeolite to be used as an adsorbent agent. The catalytic activity of zeolite as an adsorbent is

increasing with the activation of the zeolite both physically and chemically. The activated zeolite is then added to the used cooking oil to absorb impurities that have been adsorbed into the oil that has been used to fry food repeatedly.

In the purification of used cooking oil with zeolite, stirring is carried out on the sample which serves to

homogenize the mixture that has been formed. The application of stirring at a constant speed resulted in more and more impurities from the used cooking oil being absorbed by the zeolite. This can be seen through the visible physical properties of used cooking oil before and after being purified, including color, odor, and taste are shown in Figure 2.



Figure 2. Used cooking oil before being purified (a) and used cooking oil after being purified (b)

The state test of used cooking oil was carried out by comparing the physical properties of used cooking oil before and after purification by observing the physical properties of used cooking oil in the form of color, smell, and taste. The results of the observation of the condition of used cooking oil were then compared with the quality requirements of cooking oil based on SNI 01-3741-2013. From the results of the comparison of the state of the purified used cooking oil with the SNI standard, it was found that based on the physical properties observed the used cooking oil complied with SNI 01-3741-2013, namely the smell of the used cooking oil that was purified normal and not rancid, the taste of the used cooking oil that was purified was normal, and the properties of the used cooking oil. The last physical observation observed was the color of the purified used cooking oil which showed a pale yellow color.

3.3 Quality Test of Refined Cooking Oil

The quality of the purified used cooking oil was tested by measuring the peroxide number, free fatty

acid content, acid number, and water content and compared with the quality requirements of cooking oil based on SNI 01-3741-2013. The quality of used cooking oil that was purified with zeolite was compared with the quality of used cooking oil without purification.

3.4 Free Fatty Acid Level

One of the parameters that indicate the declining quality of the oil is the high number of free fatty acids caused by repeated heating. Repeated heating of cooking oil will cause hydrolysis and oxidation of cooking oil which causes the formation of glycerol and free fatty acids. The high content of fatty acids causes an unpleasant taste in cooking oil. In addition, the high content of free fatty acids will cause disease, one of which is increased cholesterol level in the blood. The results of the measurement of free fatty acid levels in used cooking oil samples before and after purification are shown in Table 2.

Table 2. Free Fatty Acid Test Results

Sample	Variation of addition of zeolite (gr)	Free fatty acid (%)	SNI 01-3741-2013 (%)
Initial sample	0	0.545	0.3
Sample 1	7	0.23	
Sample 2	8	0.23	
Sample 3	9	0.227	
Sample 4	10	0.22	

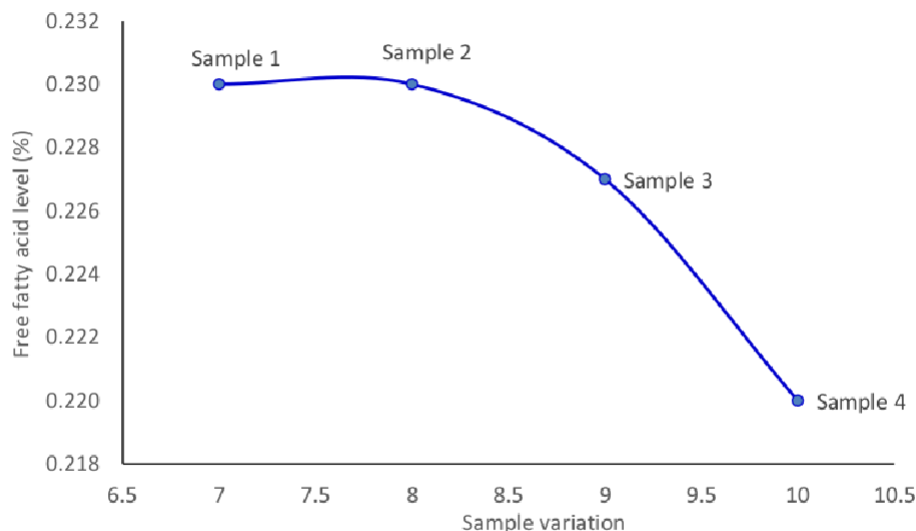


Figure 3. The relationship between variations in the addition of zeolite (gr) with free fatty acid levels (%)

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From Table 2, it is known that the fatty acid content of used cooking oil before being purified does not meet 9: SNI standard for cooking oil quality, while the used cooking oil after being purified with zeolite meets the SNI standard, which is a maximum of 0.3% for 3: variations of addition with the largest decrease in free fatty acid 7: levels with the addition of zeolite of 10 grams with a free fatty acid content of 0.22%. After the addition of zeolite, the free fatty acid content decreased and decreased with the increasing amount 3: of zeolite added. The more zeolite added, the larger the surface area of the adsorbent, so the better the absorption rate. This is shown in Figure 3.

The same thing 13: was also obtained by [19, 20, 21] who carried out the purification of used cooking oil with the adsorption process using Moringa seeds as an adsorbent. The results showed that the greater the number of Moringa seeds added, the smaller the measured free fatty acid levels.

IV. CONCLUSION

The content of Si and K elements increased after activation of the zeolite, while the content of Al, Ca, and Fe elements decreased after activation. The content of the oxide compound that is the main constituent of zeolite, namely SiO₂, has increased levels after activation, which is 69.4% before activation and increases to 82.1% after activation 3:.

The results of the state test on used cooking oil that has been purified with the addition of zeolite in the form of testing for odor, taste, and color indicate that the purified used cooking oil meets the quality requirements of cooking oil based on SNI 01-3741-eISSN: 2614-0268

2013, namely producing purified oil with 3: a normal taste, no rancid and pale yellow in color. The content of free fatty acids meets the requirements of SNI 01-3741-2013 for all variations of the addition of zeolite with the highest decreasing 7: value obtained at the addition of 10 g of zeolite with a free fatty acid content value of 0.22%.

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