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Catalytic cracking of waste frying oil using Ni-Fe/activated zeolite catalyst as a source of renewable energy

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Abstract: Used cooking oil is quite abundant household waste, and is not feasible for food. The main components of oil are triglyceride compounds which have the potential as premium substitute fuels. Activated natural zeolites can be used as heterogeneous catalysts in cracking reaction of vegetable oil. Activation and impregnation of metals in natural zeolites can increase the ability of zeolites as catalysts in the reaction of cracking of used cooking oil. This study aims to prepare and characterize the active Ni-Fe/Zeolite catalyst and test the activity of Ni-Fe/active zeolite catalysts in used cooking oil catalytic cracking. The stages of this research are (1) preparation and characterization of active Ni-Fe/Zeolite catalysts by activation and adsorption of Ni and Fe metals in active zeolites (ZAk) so that they are called Ni-Fe55/ZAk, Ni-Fe57/ZAk and Ni-Fe75 catalysts/ZAk, (2) Test the activity of Ni-Fe/ZAk catalyst on catalytic cracking of used cooking oil and identification and characterization of cracking products. The results showed that the Ni-Fe/ZAk catalyst in the form of reddish granules was 30-50 mesh in size, with the character of the face area for Ni-Fe55/ZAk, Ni-Fe57/ZAk and Ni-Fe75/ZAk catalyst respectively for the surface area was 157.900-143.823 m^2/g, the pore radius is 12.197-12.244 Å, the total pore volume is 0,096-0,088 cc/g and the Si/Al ratio is 5.985-6.162. Used cooking oil can be pierced with NiFe55 ZAk catalyst at a temperature of 450°C, the yield is 31.3%. The main components of cracking are tridecane, 1-tridecene, tetradekana, 1-heptadecene, octane, nonane, 3-hexadecene, heptadecane, 8-heptadecene and heptadecane.

Keywords: zeolite, Ni-Fe metal, catalyst, impregnation, cracking

1. Background

Indonesia as one of the countries with the highest level of consumption of cooking oil in the world. Good oil only can be used two to three times frying, the use of repeated cooking oil can have an impact on human health. Cooking oil as triglycerides is composed of long chain fatty acids [1]. Termination of the long carbon chain from oil can convert into shorter carbon compounds and can potentially be a premium substitute energy source [2, 3]. Breaking reactions or cracking of vegetable oils, activation energy is needed high enough. The decrease in activation energy can be done by adding a catalyst in the reaction. There are some catalysts to crack plants oils and their derivatives such as ZSM-5 [4] , natural zeolite/Cu [5], Zn-HZSM-5/γ alumina [6], metal oxides (Co_3O_4, KOH, MoO_3, NiO, V_2O_5, and ZnO [7], and Zn-HZSM-5.

Indonesia as a tropical country has many volcanic mountains that have the potential to produce zeolites. One of the area has a lot of zeolite rocks is Sumbermanjing Wetan area, South Malang, East Java. The chemical composition of zeolites depends on local environmental conditions such as temperature, water vapour pressure and the composition of ground water in the local location. This causes zeolite with the same colour and texture and may have different chemical compositions if they

come from different locations. Zeolites in the Sumber Manjing Wetan area contain mordenite minerals with the chemical formula Na_8 $(Al_8Si_{40}O_{96})\cdot24H_2O$ [8].

Zeolites are crystalline minerals of alumina silica tetrahydrate that formed by tetrahedral $[SiO_4]^{4-}$ and $[AlO_4]^{5-}$ which are connected by oxygen atoms [9]. Zeolites are formed from an open three-dimensional skeleton that contains canals and cavities, which are filled with cations, usually alkaline metals or alkaline earth and water molecules that can move freely. The cations in the zeolite function to neutralize the negative charge on the zeolite structure, these cations are bound weakly with the zeolite structure so that they can be exchanged with other cations. Zeolites have been widely applied as adsorbents, ion exchangers and as catalysts [8, 10-12].

The performance of zeolite as a catalyst can be improved by developing monometal active ions or bimetallic because the zeolite's metal has a dual function, in addition to the metal as a catalyst, zeolite itself is a catalyst, and a combination of both is called a bifunctional catalyst. This metal was prepared by the impregnation method, namely the development of metals in active zeolites. Active metals come from transition metals such as nickel and iron [4]. Transition metals have strong adsorption power because they have none of electron pairs on *d* orbitals and are supported by the state of the *s* orbital electrons so that the concentration becomes greater at high activity in termination and bond formation [13]. Previous study shows that activated zeolite can be used as a catalyst of cellulose degradation to glucose [8], methylene blue can be degraded by Ni-Fe-Mn oxide [14], and doxorubicin degradation to non-toxic metabolites also applied Fe-Ni metal [15].

Activated zeolite catalysts can be used to accelerate the reaction of cracking vegetable oils. Catalyst cracking reactions are a way to break down complex hydrocarbons into simpler molecules that can improve quality and also reduce the amount of residue that was produced [7]. Many complex hydrocarbons are found in organic compounds, one of which is in vegetable oils. The structure of vegetable oil is composed of three saturated and unsaturated fatty acids. The alternative use of used cooking oil is very beneficial because it can reduce environmental pollution of cooking oil waste. The reaction of cooking oil cracking to fuel is more feasible [16] where the triglyceride structure in used cooking oil is similar to that of fuel and cracking fractions more effectively by using a catalyst cracking reaction.

Potential of cooking oil waste in Malang East Java is large enough. Natural zeolite, as in South Malang has the potential as a catalyst in cooking oil cracking reactions [17]. Based on the discussion that has been carried out the purpose of this study is to determine the effect of heterogeneous catalysts in catalytic cracking of used cooking oil, as well as its constituent compounds.

2. Research Methods

2.1. Tools and Materials
The tools used in this study were pestle mortar, 30 and 50 mesh sieve sizes, analytical balance sheets, plastic containers, ovens, shakers, furnaces, a set of reflux tools, porcelain cups, crucible, stirring rod, measuring flask, measuring cup, erlenmeyer, glass chemistry, spray bottles, test tubes, percolators, stationary poles, rings, methylated lamps, three-legged, asbestos gauze, glass funnels, plastic funnels, aluminium foil, universal indicators, cracking furnaces or reactors, atomic absorption spectrophotometer (AAS-AA6200), Ostwald viscometer. The material used in this study is natural zeolite from the area of South Malang, East Java, pro analytical quality chemicals such as $FeSO_4.7H_2O$, $NiSO_4$, HF, HCl, NH_4Cl, concentrated NH_3, concentrated H_2SO_4, Na_2CO_3, red methyl indicator, $BaCl_2$, hydrogen gas, nitrogen gas, used cooking oil and distilled water.

2.2. Experiment

2.2.1. Natural Zeolite Activation. Activation began by uniformation of the zeolite size with size of 30-50 mesh. Zeolites were then washed with distilled water, dried at 150ºC and dehydrated for 2 hours at 350ºC. And then zeolite was activated chemically by adding acid. The addition of acid began with the addition of HF solution to the zeolite. A sample of 1000 g of zeolite was soaked in 2000 mL of 1% HF

at room temperature for 30 minutes. Then the zeolite was washed with distilled water until the washed water was neutral (tested with a universal indicator) then dried at 150ºC. Zeolite was soaked with 6M HCl (in a ratio of 1: 5) on a round bottom flask which had been assembled with a reflux cooling device and heated for 4 hours' temperature of 80ºC. Then wash with distilled water until the washed water was neutral and dried at 150ºC. The zeolite then soaked with 1 M NH_4Cl (1: 2) for 2 hours and stirred using a shaker. Then washed with distilled water until the washed water was neutral and dried at 150ºC. Then the zeolite was calcined at 350ºC for 2 hours to free NH_3 then calcined again at 800ºC for 4 hours. This zeolite was then called the active zeolite (ZAk).

2.2.2. Metal Impregnation in Active Zeolites. The impregnation stage of the active zeolite was conducted by preparing three erlenmeyers containing 50 grams of ZAk, each of which was added by 250 mL $FeSO_4$ solution which had been made by calculating that the impregnated by zeolite had Fe^{2+}: 5% and 7% ion concentrations. The active zeolites were soaked with $FeSO_4$ solution and then washed after 24 hours. The impregnated samples were washed with distilled water until there was no SO_4^{2-} in the washing water (proven by $BaCl_2$). Zeolites were then dried in an oven at 150ºC. The dried samples were added by $NiSO_4$ solution with Ni^{2+} ion concentration of 5% for 24 hours. The sample was then washed with distilled water until there was no SO_4^{2-} in the wash water (proven by $BaCl_2$). Zeolite was then dried at 150ºC. Metal impregnated samples were calcined with nitrogen gas at 500ºC for 5 hours and reduced with hydrogen gas at 200ºC for 2 hours at a flow rate of 10 mL/sec. In the calcination process, the catalyst sample was placed in the activation reactor and heated slowly to a temperature of 500ºC and reduced by nitrogen gas. After the temperature reaches 500ºC, it was maintained for 5 hours, followed by a reduction process by flowing hydrogen gas, so that the Ni-Fe/Zeolite catalyst was obtained. The zeolite metal that is added has a Ni:Fe ratio of 5wt%:5wt%; 7wt%:5wt%; and 5wt%:7wt% so that a catalyst called Ni-Fe55/ZAk; Ni-Fe57/ZAk; and Ni-Fe75/Zak was obtained.

2.2.3. Catalyst Characterization. Zeolite catalyst characterization stage is determination of embedded metal concentration, acidity characterization, Si/Al characterization, and BET analysis. Determination of the concentration of metal embedded in the catalyst was carried out with AAS instruments. The samples were pounded until completely smooth and taken as much as 0.3 *g* for each, then dissolved with 5 mL nitric acid. Then the sample was put into a 100 mL volumetric flask, and diluted with distilled water until the boundary mark. If the solution was not clear, 50 mL of solution was taken into a 100 mL volumetric flask and diluted again to the boundary markers, and it was measured using AAS.

Determination of acidity was carried out by means of a sample heated for 2 hours then cooled in a desiccator. Each sample was taken as much as 0.3 grams and then placed in a watch glass whose mass was known. Then the sample in the watch glass was placed in a desiccator that has been placed in a porcelain cup containing concentrated ammonia. The desiccator was closed and left for 24 hours. Then the sample was removed in the desiccator and left in open air. The watch glass containing the sample was then weighed, and the ammonia mass calculated was absorbed on the surface of the sample. Si/Al ratio characterization was carried out by analysis of Atomic Absorption Spectrophotometer in ppm. BET analysis using Nova Quntachrome 4200º BET was carried out by adsorbing N_2 gas into the zeolite sample so that the total pore volume, zeolite surface area and average pore radius can be determined.

2.2.4. Catalytic Cracking of Used Cooking Oil. Used cooking oil cracking with Ni-Fe55/ZAk catalyst; Ni-Fe57/ZAk; and Ni-Fe75/Zak was carried out by means of 5 g catalyst placed in a reactor with a temperature variation of 350ºC and 450ºC, then 300 mL of used cooking oil was heated to evaporate for 2 hours at 270ºC. Used cooking oil vapour was passed on the catalyst with the help of N_2 gas, then steam was cooled and produced by distillate resulting from cracking of used cooking oil. The resulting distillate was collected based on the cracking temperature fraction. Characterization of cracking results from used cooking oil.

2.2.5. Characterization of Cracking Products. Identification of cracking products was done by looking at the optimum results of cracking products by calculating the yield then the optimum product was analysed by GC-MS to determine the composition of the compounds found in cracking products. And

then cracking products were characterized by organoleptic test, flame tests, double bond tests, density calculations, and viscosity calculations.

2.2.6. Organoleptic Test. Organoleptic tests are carried out based on physical observations of changes that occur between reactant samples and cracking products.

2.2.7. Flash Test. The flame test was done by burning the product produced from the cracking process. A few drops of cracking products are placed on the evaporation cup, then using wooden matches, the product was burned. Positive if the product lights up when it was burned.

2.2.8. Double Bond Test. A total of 10 drops of sample are put into a test tube, added with drops of bromine solution (maximum addition of 10 drops), observed changes in the colour of the added bromine.

2.2.9. Determination of Specific Mass. Specific mass was determined by weighing an empty picnometer with an analytical balance and weighing it, then adding ± 2.5 mL of sample in the picnometer and weighing it and recording the weight. The mass of the sample type was calculated by dividing the sample weight by the picnometer volume.

2.2.10. Determination of Viscosity. Determination of viscosity was carried out to test the viscosity of product samples and reactants compared to water viscosity as a reference. Viscosity measurement was done by inserting distilled water in a viscometer and note the time needed for water to reach the tera mark. The same thing was done for the sample and the time taken to reach the tera mark was recorded.

$$\frac{\eta_1}{\eta_2} = \frac{\rho_1 \text{ x } t_1}{\rho_1 \text{ x } t_2}$$

Information:
- η_1 = sample viscosity (cSt)
- η_2 = blank viscosity (cSt)
- t_1 = sample flow time (s)
- t_2 = blank flow time (s)
- p_1 = sample density (g/mL)
- p_2 = Blank density (g/mL)

3. Results and Discussion

3.1. Natural Zeolite Activation

Physical activation of natural zeolites produces zeolites measuring 30-50 mesh. Chemical activation with the addition of HF produces a reddish yellow zeolite. The addition of HF solution was intended to dissolve impurities and free Si which attaches to the zeolite pores so that the surface area increases. Addition of HCl converts the zeolite to white. The addition of HCl was aimed at extracting Al from zeolite, so that part of Al was released from zeolite and could increase acidity. While the addition of NH_4Cl solution converts zeolite to pale white. The addition of NH_4Cl solution aims to exchange cations so that the acidic site increases by heating. Zeolites are then calcined to get a regular zeolite structure. This zeolite was then called the active zeolite (ZAk).

3.2. Metal Impregnation in Active Zeolites

The zeolite metal that was added has a ratio of Ni: Fe 5wt%:5wt%, 7wt%:5wt% and 5wt%:7wt% so that a catalyst was called Ni-Fe55/ZAk, Ni-Fe57/ZAk, and Ni-Fe75/ZAk. Ni-Fe55/ZAk catalyst in the form of slightly fade reddish granules, while Ni-Fe75/ZAk catalyst in the form of reddish granules and Ni-Fe57/ZAk catalyst in the form of bright red granules.

Table 1. Percentage adsorption of Ni and Fe Metals in Zeolites

No.	Sample	% Adsorption Ni	% Adsorption Fe
1	Ni-Fe55/ZAk	96.36	98.02
2	Ni-Fe57/ZAk	93.98	98.62
3	Ni-Fe75/ZAk	97.23	98.01

From Table 1, it is known that the catalyst with the largest percentage of Fe adsorption is in the Ni-Fe57/ZAk catalyst at 98.62wt% and the catalyst with the largest percentage of Ni adsorption is in the Ni-Fe75/ZAk catalyst which is 97.23wt%. This is because the Ni-Fe57/ZAk concentration of adsorbed Fe metal is greater as much as 7wt% while the concentration of Fe metal in Ni-Fe55/ZAk and Ni-Fe75/ZAk is only 5wt%. The same thing was experienced by the same catalyst in Ni-Fe75/ZAk in Ni metal development, where Ni-Fe75/ZAk had Ni metal concentration of 7% while Ni-Fe55/ZAk and Ni-Fe57/Zak Ni metal concentrations were only 5wt %.

3.3. Catalyst Characterization

Table 2. Catalyst characterization

Sample	Acidity (mmol/g)	Si/Al ratio	Surface area (m²/g)	Total pore volume (cc/g)	Average pore radius (Å
Ni-Fe55/Zak	4.962	5.985	157.900	0.096	12.197
Ni-Fe57/Zak	4.353	6.726	169.389	0.103	12.205
Ni-Fe75/Zak	3.100	6.162	143.823	0.088	12.244

Table 3. The result of elemental analysis of zeolite before and after impregnation using XRF

Sample	Si(%)	Al(%)	K	Ca(%)	Ti(%)	V(%)	Cr(%)	Mn(%)	Fe(%)	Ni(%)	Cu(%)	Zn(%)	Sr(%)	Other(%)
Before impregnation	48.70	6.80	3.51	12.0	2.14	0.03	0.07	0.25	2.10	2.01	0.45	0.17	1.20	20.67
Ni-Fe55/ZAk	48.13	6.12	3.44	10.30	1.70	0.03	0.05	0.19	5.24	5.33	0.33	0.12	0.90	18.12
Ni-Fe57/ZAk	47.10	6.11	3.24	10.17	1.67	0.01	0.04	0.16	7.13	5.24	0.30	0.11	0.88	17.84
Ni-Fe75/ZAk	47.11	6.02	3.32	10.22	1.69	0.01	0.04	0.15	5.20	7.15	0.31	0.10	0.87	17.81

According to the Table 2, it is known that the Ni-Fe55/ZAk catalyst has the highest acidity, which is 4.962 mmol/g, this is due to the distribution of Ni and Fe metals evenly so as to increase the Lewis acid site and acidity higher. The presence of metals attached to zeolites also causes the amount of Al to decrease and increase the Si/Al ratio. The Ni-Fe57/ZAk catalyst has the largest Si/Al ratio of 6.7260. It is also known that the Ni-Fe57/ZAk catalyst has the largest surface area of 169.389 m²/g. This is due to the Ni-Fe57/ZAk catalyst being carried by Fe metal which has the highest concentration of 7wt%, where the presence of metals having small radius is able to be distributed evenly into the cavity of zeolites to form new aggregates, the presence of metals in the zeolite cavity causes the pore fingers to grow smaller causing the catalyst surface area to increase. The existence of new aggregates formed by the presence of metals in zeolite is proven by the smaller Ni-Fe57/ZAk catalyst radius and the large total pore volume, which is 0.103 cc/g.

Table 3 shows that metals of Fe and Ni was success to impregnated in the catalyst of natural zeolite. Percentage of both metals are nearly considered that is between 5 to 7 wt%. Therefore, these catalyst is then used for cracking cooking oil.

3.4. Catalytic Cracking of Used Cooking Oil

Cracking is done for 2 hours with used cooking oil as a reactant. Cracking products are clear yellowish liquid. The resulting cracking products are accommodated in a container to measure the volume and calculate the yield. So that data is obtained in Table 4.

Table 4. Catalyst characterization

Sample	Cracking temperature (°C)	Product name	Volume (mL)	Yield (%)
Without catalyst	450	Thermal cracking	36	12.0
Ni-Fe55/ZAk	350	Sample 1	86	28.7
Ni-Fe57/ZAk	350	Sample 2	81	27.0
Ni-Fe75/ZAk	350	Sample 3	84	28.0
Ni-Fe55/ZAk	450	Sample 4	94	31.3
Ni-Fe57/ZAk	450	Sample 5	91	30.3
Ni-Fe75/ZAk	450	Sample 6	89	29.7

In Table 4, it is known that thermal cracking without catalyst results in a lower yield of 12wt% compared to cracking using a catalyst. The existence of an active Ni-Fe/Zeolite catalyst is able to accelerate cracking reactions as evidenced by the number of cracking products that are more produced for 2 hours compared to non-catalyst thermal cracking products. This proves that the zeolite that put on by metal is able to act as a catalyst [18].

In Table 4 it is also known, that sample 4 from Ni-Fe55/ZAk catalyst at 450ºC produces the most cracking product which is 94 mL. And from the calculation of the yield, it was obtained 31.3wt%. This can be seen because sample 4 uses Ni-Fe55/ZAk catalyst which is carried by the same Ni and Fe metal (5wt%:5wt%), where the presence of Ni and Fe metals spread evenly in the zeolite cavity can increase acidity so that its ability as an optimum catalyst. This is supported by a large surface area and small pore radius.

3.5. Identification and Characterization of Cracking Products

Identification of cracking products in sample 4 using GC-MS resulted in a chromatogram seen in Fig. 1.
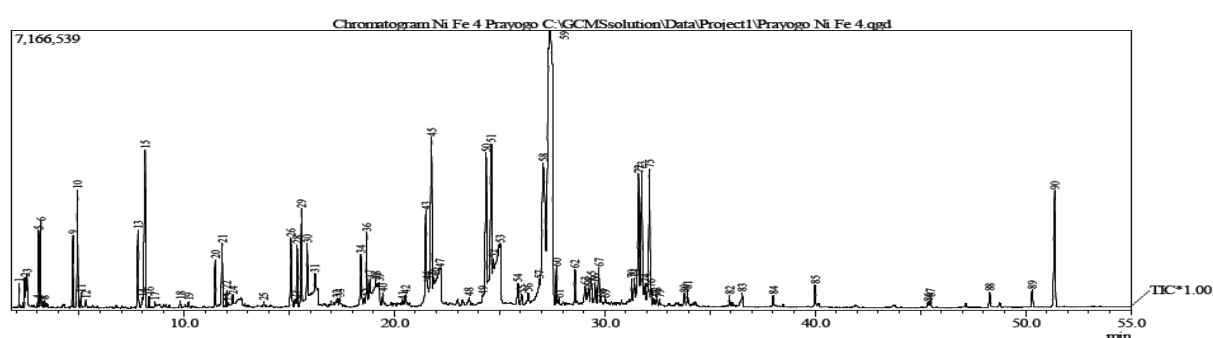
Figure 1. Chromatogram of Sample Cracking Products 4.

Figure 1 shows, there are several constituent compounds of cracking products with retention time and area width of each. From the several peaks, 10 peaks were chosen which had the largest area. Retention time and area of compound of cracking products are listed in Table 5.

Table 5. Retention Time and % area of each compound as an oil cracking products.

R. time	area	% area
21.767	15885114	3.67
27.067	33270983	7.69
27.383	107749371	24.89
31.617	16787252	3.88
4.932	8266423	1.91
8.150	14810344	3.42
24.367	15002841	3.47
24.592	14127046	3.26
31.758	16778043	3.88
32.125	14272397	3.30

Based on the data GC was obtained the percentage of constituent compound as listed at Table 4. By using Gas Chromatography-Mass Spectrometry (GC-MS), it can be found the name of each constituent compound of cracking products and the main compounds are tridecane (3.67%), 1-tridecene (7.69%), tetradecane (24.89%), 1-heptadecene (3, 88%), octane (1.91%), nonane (3.42%), 3-hexadecene (3.47%), heptadecane (3.26%), 8-heptadecene (3.88%) and heptadecane (3.30%). This result in line with the study conducted by Budianto et.al. shows that cracking plant oil can produce long chain hydrocarbon [4, 6].

3.6. Characterization of Cracking Products

Table 6. Characteristics of Sample Cracking Products No. 4.

Parameter	Using cooking oil	Product
Organoleptic Test	Yellowish brown Smells like coconut oil	Coloured liquid Yellowish and stinging
Burning Test	Doesn't burnt	Burnt
Double bond Test	Positive there are double bonds	Positive there are double bonds
Density (g/mL)	0.7924	0.6721
Viscosity (cSt)	62.8722	2.2762

From Table 6, it is known that the organoleptic test showed different results between used cooking oil and cracking products. The form of used cooking oil and cracking products is different where cooking oil is in the form of thick brown liquid while cracking products are clear yellowish. From odour identification and combustion tests it can also be proven that cracking products are new compounds, no longer as cooking oil. Cracking products smell stinging and can burn while cooking oil smells like coconut oil and does not burn. This means that the compounds contained in used cooking oil are different from compounds in cracking products.

Test the presence of double bonds gives positive results on cracking products, characterized by the fading of bromine solution so that it can be concluded that cracking products contain double bonded hydrocarbons or alkene. Alkene according to GC-MS results is derived from 1-tridecene and 8-heptadecene compounds.

From the determination of viscosity, used cooking oil has a viscosity of 62.8722 cSt, far greater than the viscosity of the sample 4 cracking product of 2.276 cSt. This means that the presence of an active Ni-Fe/Zeolite catalyst has been successfully used to remove used cooking oil into a new, thinner compound which is proven by low viscosity. According to Decree of the Director General of Oil and Gas No: 3675 K/24/DJM/2006 diesel oil viscosity is 2.0-5.0 cSt, and cracking products have viscosity that is in that range, so it can be said that cracking products have the potential as alternative fuels.

4. Conclusion

The results showed the active Ni-Fe/zeolite catalyst in the form of reddish granules measuring 30-50 mesh. Characterization of Ni-Fe55/ZAk, Ni-Fe57/ZAk and Ni-Fe75/ZAk catalysts for surface area were 157.900; 169.383; 143.823 m^2/ g, the pore radius is 12.197; 12.205; 12.244 Å, the total pore volume is 0.096; 0.103; 0.088 cc/g, the amount of acidity is 4.962; 4.353; 3.100 mmol/g, and the Si/Al ratio is 5.985; 6.726; 6.162. Ni-Fe/ZAk catalyst successfully used cooking oil into cracking products, with optimum cracking results with a yield of 31.3wt% using NiFe55/ZAk catalyst at 450ºC and the composition of the cracking products was tridecane (3.67wt%), 1-tridekena (7.69wt%), tetradecane (24.89wt%), 1-heptadecene (3.88wt%), octane (1.91wt%), nonana (3.42wt%), 3-hexadecene (3.47wt%), heptadecane (3.26wt%), 8-heptadecene (3.88wt%) and heptadecane (3.30wt%).

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