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Used cooking oil catalytic cracking using Cr-charcoal ion-exchanged catalyst

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Abstract. Used cooking oil is the largest waste that can be converted into biofuel as a renewable alternative energy source with catalytic cracking process. In this study, used cooking oil was cracked using a Cr-Carbon catalyst. The charcoal was produced from solid waste (shell) of oil palm industry. Three different chromium concentrations (1, 2, and 3%) were used for the ion-exchange process of Cr-charcoal to produce the catalyst. These catalysts were used for the catalytic cracking of used cooking oil at three levels of temperature (450, 500, and 550°C). X-ray diffraction (XRD) and scanning electron microscopy (SEM) analysis were used for catalyst characterization. SEM-energy dispersive X-ray spectroscopy (EDX) analysis indicates that Cr was successfully adsorbed into the catalyst. The highest oil fraction was obtained from catalytic cracking of used cooking oil at 450°C using Cr-charcoal catalyst synthesized using 1% Cr solution.

1. Introduction

The depletion of fossil fuel resources and the environmental issue of fossil fuel using have become public concern at present time. Fossil fuel cannot be renewed and has limited existence. On the other hand, the world energy consumption is increasing and causes the fuel resources diminished. This leads scientist over the world to discover alternative fuels based on renewable material. Bio-fuel is an alternative energy that can be developed in Indonesia as an alternative for fossil fuel due to its renewable, non-toxic and biodegradable properties [1, 2].

The cost of bio-fuel production for commercialization is not economical in comparison to fossil fuel. Used cooking oil is one of the economical sources for bio-fuel production. Used cooking oil contains water and free fatty acids. Used cooking oil is defined as used vegetable oil obtained after cooking food and not suitable for use and reuse can be harmful for our health. Used cooking oil pollute the environment as its presence in water can inhibit oxygen exchanges and damage ecosystems. The application of used cooking oil as raw material for bio-fuel production can reduce pollution of water, reduce the cost of bio-fuel production, greenhouse gas reductions and increase economic value of used cooking oil [1-3].

Cracking process is the solution to recycling of used cooking oil into fuel. It is widely used process in petroleum refining for converting heavy portion into lighter products. Cracking process can be done by two processes: thermal cracking and catalytic cracking process. Thermal cracking is a simple process



where heat is used to cut long hydrocarbon chain. In catalytic cracking, long carbon chains are broken into shorter and simple hydrocarbon chains or molecules with the help of a catalyst. The catalyst reduces the activation energy of the reaction to accelerate chemical reactions, controls the selectivity of the product and improve product quality and quantity [3-8].

Key properties that influence the catalytic activity in the catalytic cracking reaction are acidity, size, pore shape, and selectivity. Furthermore, it is stable at high temperatures and easily separated from the product. Heterogeneous catalysts which consisted of metal with active materials and metal-supported catalysts are catalysts usually used for this cracking process. Activated carbon is widely used for catalyst carrier, because it is stable in acidic and alkaline environments. Activated carbon can be produced from coconut shell. Coconut shell is a good material for catalyst carrier due to its large micro pores, low ash content, and high reactivity [9-12].

Impregnation, ion exchange, adsorption, and deposition-precipitation are some methods that can be used for catalyst preparation for the cracking process. Impregnation is performed by immersing the catalyst carrier in a solution that contains a precursor salt with the active metal. In such case, the carrier provides a large surface so the impregnation process will be more efficient. Impregnation is achieved when there is no anion or cation exchanged with the active phase. Contrary to the impregnation method, in this research, the process of preparation catalyst using ion-exchange method where chromium metal (Cr) used as an active catalyst site which is placed on activated charcoal. The use of activated carbon as catalyst carrier of the active metal can increase the selectivity of the catalyst. The principle of ion exchange method is to interchange ions from the carrier with the active metal. This method is carried out by inserting cations into zeolites through the exchange of alkali or alkaline earth cations using salts precursor [3, 8, 13-17].

The objectives of this study were to determine the characteristic of Cr-charcoal catalyst produced by ion exchanged using several concentration of Cr-solution and to determine the performance Cr-charcoal catalyst in the cracking of used cooking oil into fuels.

2. Methodology

2.1. Materials

The materials used in this study were used-cooking oil was acquired from household waste, charcoal from palm oil shell, sodium carbonate, acetic acid, chromium tri-nitratenanohydrate, filter paper, litmus paper and distilled water.

2.2. Instrumentation

Analytical instrumentations used in this study were distillation equipment, cracking reactor (stainless steel, developed from the model of Nazarudin [6] was used for the catalytic cracking, Gas Chromatography, XRD, SEM provided by ZEISS version EVO MA 10 and other laboratory instrumentation such as oven, hot plate, magnetic stirrer, analytic balance, vacuum pump, porcelain glass, and measurement glass.

2.3. Procedure

2.3.1. Sample preparation

Used cooking oil from used 3 times used household frying oil was filtered using a method investigated by Nazarudin [18] where the adsorption device was constructed from a 2 inch pipe and the adsorption media was charcoal from kernel palm shells.

2.3.2. Catalyst production process

There were two steps in the catalyst synthesis (1) charcoal activation and (2) catalyst modification using Cr. The Charcoal activation followed the method of Nazarudin[13]. The Charcoal activation included preparation of raw materials (sodium carbonate, distilled water and the charcoal) and weighed according to the data using 1:1:1 ratio were mixed and stirred for 2 hours at room temperature. Then the mixture was washed with distilled water while filtered using a vacuum pump until it reached neutral pH and heated into the oven at 105°C for 4 hours. After that, the catalyst was activated with steam at 550°C for 5 hours.

The catalyst was modified using a procedure of Nazarudin[17]. The Chromium Trinitrate Nano-hydrate solutions were prepared with various concentrations, 1, 2, and 3%. Activated charcoal and Cr metal solution were mixed (1:10 ratio) and stirred for 24 h at room temperature. The solution was then washed to reach pH 7 and filtered. The residue was dried in the oven at 105°C for 24 h.

2.3.3. Catalytic cracking process

Used cooking oil was placed inside the reactor, which was then filled with different concentrations of the catalyst 1, 2, and 3%. Nitrogen gas was flowed during the catalytic cracking process. The output of the reactor was poured into an ice trap for cooling. The cracking process was carried out at different temperatures: 450, 500, and 550°C for 75 min. Product from the cracking process taken every 15 min.

3. Result and discussion

3.1. X-Ray Diffraction (XRD) Analysis

X-ray Diffraction (XRD) analysis is a method used to identify the crystalline phase in material and to get the size of the particles. This analysis can provide qualitative and semi-quantitative data on solids or samples. Figure 1 shows the diffraction pattern of the carbon catalyst with and without chromium. The peaks in $2\theta = 21.8791^\circ$; 20.7753° ; 21.7491° for 1% Cr-metal solution, 2% Cr-metal solution and 3% metal solution. Based on the above diffraction pattern, indicates that all catalyst has the same pattern.

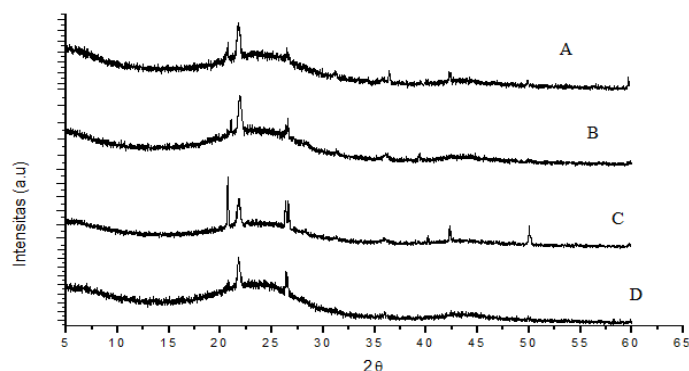


Figure 1. XRD patterns of carbon catalyst: (a) without Cr and after ion exchange with (b) 1, (c) 2, and (d) 3% Cr

3.2. Scanning electron microscopy and energy-dispersive X-ray spectroscopy (SEM-EDX) analysis

SEM-EDX is used to find out and describe the morphology and content of the catalyst. The results of SEM-EDX analysis modified catalyst can be seen in the figures 2-3. Based on figures 2-3, it can be seen that the morphology of the catalyst before and after modification with zooming 1000x, has a form of aggregate with nanoparticle size. The figure shown that the activated charcoal has cavities or pores with 1 μm distance. The largest area of pore was 17.27, 17.27, and 95.36 μm^2 for the 1, 2, and 3% Cr concentrations. Table 1 shown that the catalyst contains where Cr-metal was successfully impregnated into the activated charcoal, this is indicated by the presence of Cr content in the modified activated

charcoal. The higher of Cr content is in 2% Cr-metal concentration. The purpose of added active metal on catalyst surface is increased the activity of the catalyst. So that increasing of yield of gasoline.

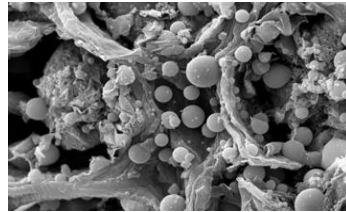
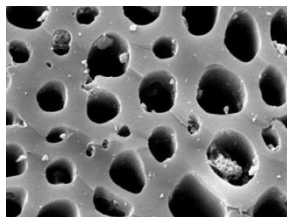
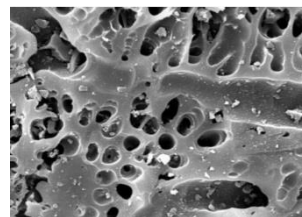


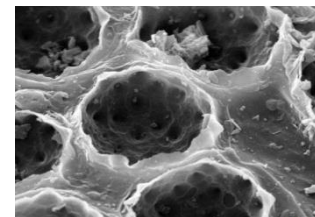
Figure 2. SEM image of activated palm shell charcoal



(a)



(b)



(c)

Figure 3. SEM image of activated palm shell charcoal after ion-exchange treatment with (b) 1, (c) 2, and (d) 3% Cr

Table 1. Elemental content of the activated charcoal before and after modified with 1%, 2% and 3% Cr-metal

Elements	Content (%-w)			
	Before modified	After modified with Cr-metal		
		1%	2%	3%
C	62.32	96.33	91.93	94.92
Si	35.35	2.86	4	2.14
P	2.14	0.5	1.75	2.11
Ca	0.2	0.05	0.11	0
Mg	0	0.14	0.95	0.07
Al	0	0	0.12	0
Cr	-	0.12	1.15	0.76

3.3. Effect of temperature and catalyst content on the cracking process

The process was carried out both thermally and catalytically. Thermal and catalytic cracking processes were carried out at 450°C, 500°C, and 550°C for 60 minutes. The cracking process was carried out in a batch reactor by placing the catalyst into the bed in a vertical reactor and the raw material into the horizontal reactor. Table 2 shows that the temperature affects the percentage of cracking liquid yield. The higher of the temperature at thermal cracking, the more liquid yield produced. It shown that at the temperature 550°C the cracking liquid yield was 52.34% higher than temperature 450°C and 500°C.

Table 2. The result of thermal and catalytic cracking of used cooking oil using various Cr-charcoal concentrations

Cr-solution Concentration	Temperature (°C)	Used cooking oil (gr)	Liquid product (%)	Gas yield (%)	Residue (%)
Thermal	450	52.72	35.60	-	-
	500	52.09	47.14	-	-
	550	53.13	52.34	-	-
1%	450	23.00	45.22	48.48	6.30
	500	23.55	14.10	84.50	1.40
	550	21.16	29.02	69.00	1.98
2%	450	23.87	14.79	84.79	0.42
	500	20.90	9.38	89.76	0.86
	550	23.49	21.50	77.99	0.51
3%	450	23.83	14.94	84.73	0.34
	500	21.52	34.90	61.20	3.90
	550	22.75	19.65	80.35	0

Table 3. GC-MS of the liquid product from catalytic cracking of used cooking oil using 1% Cr-ion exchanged charcoal

No	Number of Carbon	% Area	Molecular Weight
1	C3	18.33	58
2	C7	0.99	122
3	C8	4.32	110-114
4	C9	1.13	128
5	C10	0.36	140
6	C11	45.62	154-156
7	C12	10.84	168-170
8	C17	5.42	98-270
9	C20	12.98	310

3.4. Gas chromatography-mass spectrometry (GC-MS) analysis

GC-MS is used to identify different compounds within a test sample. The GC-MS results show that used cooking oil contained carbon compounds with chains from C3 to C20 with molecular weight ranging from 58 to 310 (Table 3). Based on the GC-MS results, the largest product obtained from the catalytic cracking process using 1% Cr-charcoal was diesel oil with total C8 – C20 was 80.67%. The temperature and type of catalyst affected the total conversion and selectivity of the results of the catalytic cracking reaction. The yields of hydrocarbon in catalytic cracking depend on the choice of shape selective catalyst as well as acidity of the catalysts.

4. Conclusion

In this study, we have developed catalyst with ion exchange modification for catalytic cracking of used cooking oil to produce an alternative non fossil fuel source. Cr-charcoal catalyst can be an alternative catalyst for the catalytic cracking of used cooking oil. The purpose of active metal addition on catalyst surface was to increase the active side on the catalyst surface, so the activity of the catalyst increased. Both temperature and concentration of Cr-solution in the production of catalyst influenced the catalytic

cracking process. The highest yield for the oil fraction was obtained from cracking of used cooking oil at 450°C with the Cr-charcoal synthesized using 1% Cr solution. Using this catalyst, the product was mainly diesel oil (C8–C20) with percent area 80.67%.

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