



Catalytic Cracking of Crude Palm Oil Using Ni-Carbon with Ion Exchange Method

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Abstract: According to data from the National Energy Council (DEN) (2016), the energy consumption in Indonesia is increasing every year and will increase 1.8 times in 2025, this requires an alternative energy to back up the energy needs. One such alternative energy strategy is to use biodiesel. Biodiesel can be obtained from Crude Palm Oil using a catalytic cracking process. This study investigated the cracking of Crude Palm Oil using Ni-carbon with ion exchange method. The concentration of the metal solution used was 1, 2 and 3%, with temperature 450, 500 and 550 ° C. The catalyst was analyzed by XRD and showed that the highest pattern at 25.6° on 2% solution. The catalyst characterized by SEM-EDX showed that the nickel metal bonded to the 1, 2, and 3% Ni-Charcoal catalysts was 02.01, 1.13 and 2.09%, respectively and the average of catalytic cracking product conversion at concentrations of 1, 2 and 3% was 62.55, 66.52 and 56.50%, respectively.

Keywords: Biofuel; Crude palm oil; Cracking

Introduction

Fuel Oil is one of the human needs that continues to increase along with the yearly increase of population. According to Special Task Force for Upstream Oil and Gas (SKK Migas), which is the special task force for upstream oil and gas activities, Indonesia's total oil reserves as of December 31, 2021 were 2.36 billion barrels, a decrease compared to conditions on January 1, 2020, which was 2.44 billion barrels. In general, the availability of national fossil energy in 2018 was 7.51 billion barrels, decreased by 0.27% compared to 2017. Likewise, natural gas reserves decreased by 5.02% from 2017 (BPS, 2021).

Indonesia has great potential in the palm oil sector. In 2019, the area of oil palm plantations in Indonesia reached 14.68 million hectares. Palm oil mills have become a leading agricultural commodity by producing 27,898,875 tons Crude Palm Oil (CPO) (BPS, 2021). Long chains of hydrocarbons contained in crude palm oil can be broken down into shorter chains through a cracking

process to produce biofuels. One of the biofuels produced from palm oil is biodiesel. Biofuels are defined as mono alkyl-ester chains of fatty acids derived from animal and vegetable acids. Fatty acids of vegetable origin can be used as a source of renewable, non-toxic and biodegradable biofuels (Canakci, 2001, Sabagh, 2011; Sirajudin et al., 2013; Nazarudin et al., 2017; Nazarudin et al., 2007a).

There are two types of cracking processes, (1) thermal cracking and (2) catalytic cracking. Thermal cracking is a simple process but requires high energy, which is used to breaks down a long hydrocarbon chains (Nazarudin, 2000; Lin et al., 2013; Li et al., 2014; Luo et al., 2010). While in catalytic cracking, long-chain hydrocarbons break down by using a catalyst (Roesyadi et al., 2013; Yigezu & Muthukumar, 2014). The used catalyst is a catalyst that is stable at high temperatures and is easily separated from the cracking products.

Activated carbon is widely applied in the catalytic cracking process because it has a large adsorption capacity and is stable in both acidic and alkaline

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conditions (Prabasari et al., 2019; Riyanto et al., 2017). Activated carbon derived from palm oil shells has advantages because of its low ash content, reactive and large pore size (Nazarudin et al., 2019; Pickworth & Trueblood, 2010). As a catalyst carrier, nickel metal is quite widely used because it is relatively inexpensive and has good activity and selectivity, furthermore nickel metal has the advantage that the active components of the metal can be distributed evenly throughout the surface of the carrier pores to increase the surface area of the catalyst, has high stability and great resistance to sintering (Zhang et al., 2021; Lestari, 2012; Pilbanjahromi et al., 2013). Nickel metal has an open plane crystal structure which can break C-C and CH bond (Harjito, 2012; Kolasinski, 2022). In addition, the selection of Nickel metal is also based on the characteristics of Nickel metal which has an unfilled d orbital, this causes Nickel metal to have the opportunity to form bonds with other elements (Sari et al., 2017; Nurhayati & Wigiani, 2014). Nickel catalysts also has a low cost, some studies have been carried out on preparation of nano-nickel catalysts supported on different low-cost (Selim et al., 2009; Selim & Fathy, 2012; Du & Chen, 2007).

The aim of this study was to determine the diffraction pattern of activated charcoal catalyst modified with nickel metal, the effect of embedding nickel metal concentration on the conversion of cracking results, as well as the density of the cracked liquid compared to the density of commercial fuel oil on the market.

Method

The materials used in this study were crude palm oil (CPO) from PTPN VI, activated charcoal made from palm oil shells, Ni(NO₃)₂·9H₂O, NaOH, and distilled water. The research stages consisted of, (1) raw material preparation, (2) catalyst manufacture including activation process of activated carbon, preparation of Ni(NO₃)₂·9H₂O solution, and ion exchange process, (3) cracking process, and (4) analysis.

Preparation of samples

Preparation of raw materials was carried out by weighing 50 kg of CPO.

Catalyst production process

The catalyst manufacturing process began with the activation of activated carbon made from palm oil shells. Charcoal was activated with a solution of NaOH and distilled water in a ratio of 1: 1: 3, the mixture was stirred for 2 hours at room temperature, then washed with distilled water. Then soaked with the addition of 5 ml of vinegar for 30 minutes, and washed again using distilled

water to get a pH of 7. The activated charcoal was baked at a temperature of 105°C for 4 hours and then steamed using a steam reactor with a temperature of 550°C for 6 hours. The next step was making a solution of Ni(NO₃)₂·9H₂O. This solution was made with variations of 1, 2, 3% using 100 ml of distilled water.

The catalyst modification process was carried out by the method introduced by Nazarudin (Nazarudin et al., 2007b). The ion exchange process was carried out by mixing activated charcoal with a solution of Ni(NO₃)₂·9H₂O in a ratio of 10:1 for 24 hours at room temperature. The solution was washed to pH 7, then filtered to obtain 2 layers, namely the filtrate and residue. The filtrate was then separated from the residue and dried using an oven for 12 hours at a temperature of 105°C to obtain a Ni-carbon catalyst with variations in Ni solution 1, 2 and 3%. Catalyst characterization was carried out using X-ray Diffraction (XRD) and scanning electron microscopy-energy X-ray (SEM-EDX).

Catalytic Cracking Process

The cracking process was carried out in a semi-batch in a cracking reactor with a catalyst ratio and raw material of 1:10, the cracking temperature varied at 450, 500, and 550 °C for 120 minutes.

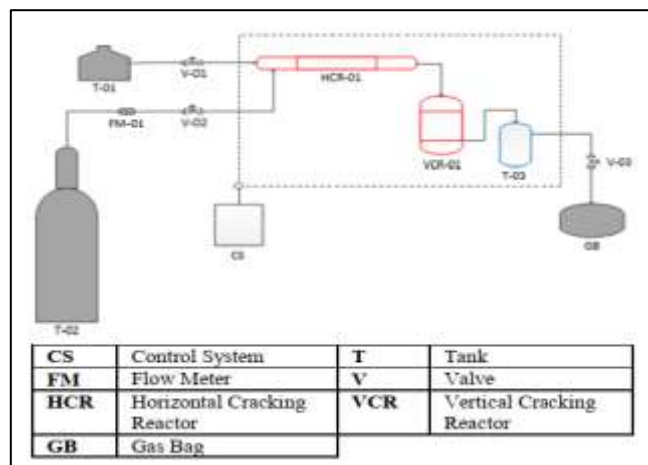


Figure 1. Schematic representation of the experimental semibatch reactor setup

Result and Discussion

Catalyst characterization

X-ray Diffraction (XRD) analysis was performed to identify the crystalline phase and the amorphous phase of the catalyst. The following are graphs of XRD analysis results for pure Carbon catalysts and Carbon that have been modified by adding Ni as carrier metal with a concentration of 12 and 3%.

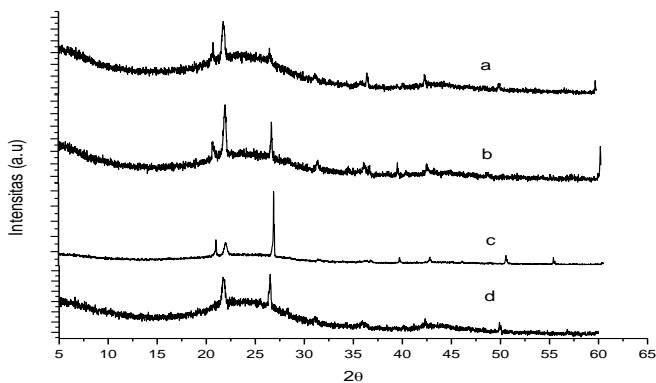


Figure 2. XRD diffraction schematic (a) pure carbon, (b) Ni-Carbon 1%, (c) Ni-Carbon 2%, and (d) Ni-Carbon 3%

The diffraction pattern in Figure 2 showed that all peaks on the carbon catalyst have a fairly high intensity at $2\theta = 21.8^\circ; 21.9^\circ; 26.6^\circ; \text{ and } 26.5^\circ$. The Ni/C diffraction pattern is shown at angles of $2\theta = 26.6^\circ \text{ and } 26.5^\circ$. The diffraction pattern shows Nickel metal embedded into the activated charcoal catalyst.

Figures 2 and 3 showed the results of scanning electron microscopy-energy X-ray (SEM-EDX) analysis which describe the surface morphology of the catalyst under conditions before modification and after modification using 1, 2 and 3% nickel metal.

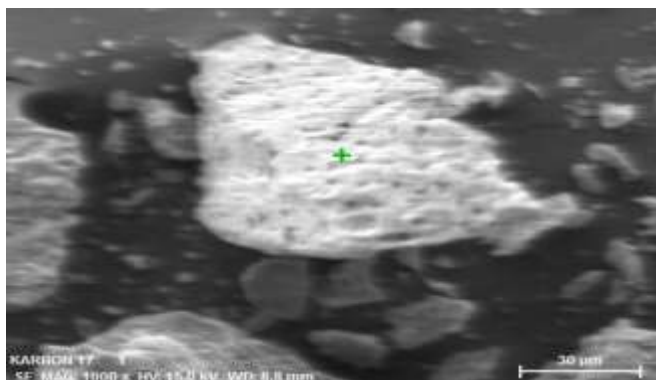
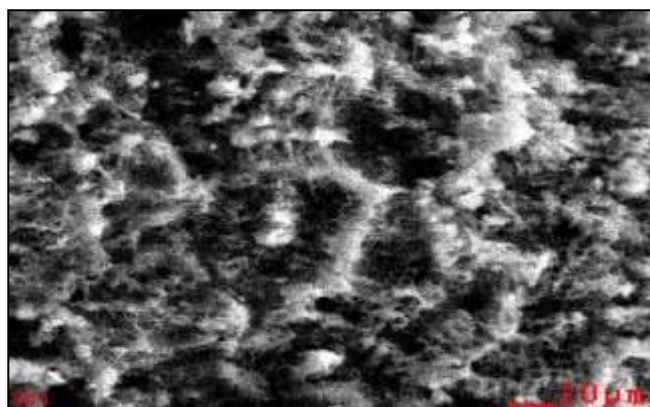
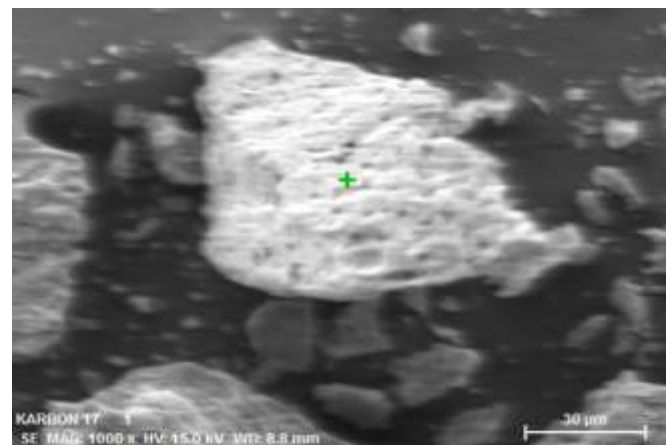


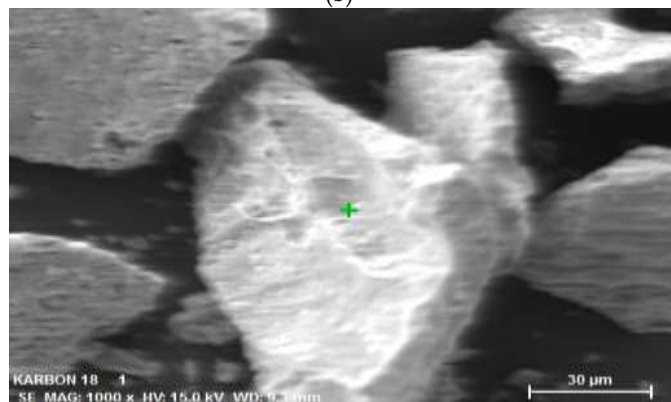
Figure 3. Microstructure of activated carbon



(a)



(b)



(c)

Figure 4. Microstructure of activated carbon after modification with Ni at different concentrations. (a) 1% Ni, (b) 2% Ni, and (c) 3% Ni

Effect of cracking process temperature and Ni concentration

The cracking process was conducted both thermally and catalytical. Thermal cracking was performed at 450, 500, and 550 °C whereas catalytic cracking was performed at the same temperatures with different Ni-carbon catalysts. Table 1 summarizes the results of the thermal cracking process.

Table 1. Thermal cracking process results

| Temp (°C) | CPO (gr) | % Conversion | | |
|-----------|----------|--------------|-------|---------|
| | | Liquid | Gas | Residue |
| 450 | 94.44 | 55.06 | 41.16 | 3.78 |
| 500 | 99.94 | 57.97 | 37.19 | 4.83 |
| 550 | 100.00 | 60.10 | 38.95 | 0.95 |

The results showed that the raw materials are converted into liquids where the temperature affects the amount of conversion. The higher the temperature was, the more conversion percentage obtained, which is 60.10% at a temperature of 550°C.

The catalytic cracking process was carried out using an unmodified carbon catalyst and a modified Ni-carbon catalyst with various concentrations of Ni solution (1%, 2% and 3%), the cracking process was carried out at temperature variations 450, 500 and 550°C to each

catalyst. The ratio of raw materials and catalysts used in this process was 1:30. Table 2 below showed the conversion of catalytic cracking results using unmodified carbon catalyst.

Table 2. Conversion of catalytic cracking results using unmodified carbon catalysts

| Temp (°C) | CPO (gr) | % Conversion | | |
|-----------|----------|--------------|-------|---------|
| | | Liquid | Gas | Residue |
| 450 | 12.51 | 72.45 | 25.19 | 2.36 |
| 500 | 114.61 | 68.88 | 28.29 | 2.83 |
| 550 | 101.45 | 64.85 | 34.26 | 0.89 |

The result showed that the highest conversion of 72.45% was obtained at a temperature of 450°C, this catalytic process conversion was higher than thermal cracking. This showed that the catalyst can help to achieve the high conversion in the cracking process without using high temperatures.

Conducted by Zhang et al (2021), the catalytic cracking process of tar biomass at various temperatures of 600, 700 and 800°C using Ni with various concentrations of 0.15, 0.30, and 0.60 mol/L which was added to the carbon nanofiber and porous carbon catalyst (Ni@CF/PCs) showed that the total pore volume of Ni@CF/PCs would increase with increasing concentration of Ni metal. Meanwhile the conversion reached 95.54% at 800°C using 0.30 mol/L Ni metal concentration and 94.78% at 700°C using the same Ni metal concentration. This showed that there is an effect of the concentration of metallic Ni which is deposited to open the pores on the catalyst and increase the cracking efficiency (Zhang et al., 2021; Mahmudah & Hari Kusumawati, 2020; Ramadhani et al., 2017). (Endarko & Adawiyah, 2019). Table 3 showed the results of catalytic cracking using modified catalysts with 1, 2, and 3% Ni concentration.

Table 3. Conversion of catalytic cracking results using Ni-Carbon catalyst with various concentration

| Temp (°C) | CPO (gr) | % Conversion | | |
|---|----------|--------------|-------|---------|
| | | Liquid | Gas | Residue |
| Ni-Carbon catalyst with 1% Ni concentration | | | | |
| 450 | 104.07 | 68.25 | 27.98 | 3.77 |
| 500 | 103.13 | 60.04 | 37.68 | 2.28 |
| 550 | 109.70 | 67.36 | 32.11 | 0.53 |
| Ni-Carbon catalyst with 2% Ni concentration | | | | |
| 450 | 125.84 | 74.14 | 24.00 | 1.84 |
| 500 | 110.97 | 64.22 | 33.63 | 2.15 |
| 550 | 100.65 | 61.21 | 37.22 | 1.57 |
| Ni-Carbon catalyst with 3% Ni concentration | | | | |
| 450 | 108.21 | 63.48 | 30.83 | 5.69 |
| 500 | 107.34 | 66.67 | 31.11 | 2.23 |
| 550 | 110.66 | 39.35 | 58.49 | 2.16 |

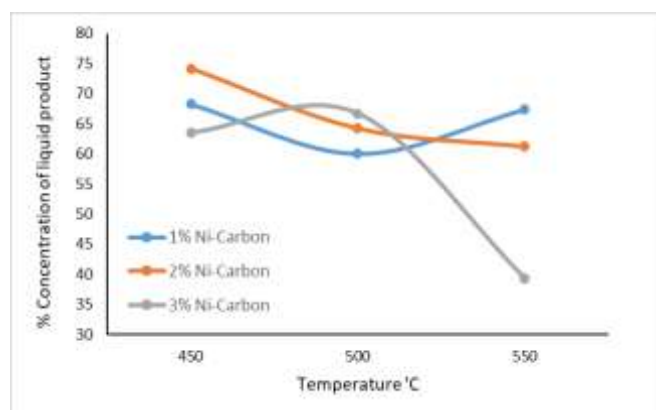


Figure 5. Conversion percentage of liquid product vs temperature for catalytic cracking

Density analysis

Density analysis was carried out as one of the parameters to compare the liquid cracking product with commercial liquid fuels. The maximum density value for commercial diesel/bio-solar fuel is 0.860 gr/ml, and the density of gasoline is in the range of 0.715 to 0.770 gr/ml. The results of the density analysis obtained from the catalytic cracking process using 1, 2 and 3% Ni-carbon catalysts are shown in Table 4.

Table 4. Density analysis

| Ni-Carbon Concentration | Temp (°C) | Density of liquid product (gr/ml) |
|-------------------------|-----------|-----------------------------------|
| 1% | 450 | 0.91846 |
| | 500 | 0.91772 |
| | 550 | 0.94188 |
| 2% | 450 | 0.95142 |
| | 500 | 0.91974 |
| | 550 | 0.95276 |
| 3% | 450 | 0.94148 |
| | 500 | 0.93132 |
| | 550 | 0.94352 |

Table 4 showed that the density value of the cracking liquid was in the range 0.91772 – 0.95276 gr/ml, lower than crude palm oil density as raw material, the density of crude palm oil is 0.988 gr/ml. It indicated that long-chain hydrocarbons in crude palm oil breaks down and results more carbon chains.

The density value for commercial liquid fuels on the market based on data from the National Standardization Agency and Pertamina in 2020 showed in Table 5 (Pertamina, 2020).

Table 5. Density value for commercial liquid fuels

| Fuel Oil | Density (gr/ml) |
|-----------------|-----------------|
| Solar/Bio-solar | Max 0.860 |
| Kerosene | Max 0.835 |
| Gasoline | 0.715 – 0.770 |
| Fuel Oil 380cSt | Max 0.991 |
| Diesel Oil | Max 0.920 |

From data shown in Table 5, it can be seen that the liquid resulting from catalytic the cracking process using a Ni-carbon catalyst is in the range of fuel oil and diesel oil.

Conclusion

X-ray Diffraction (XRD) analysis showed that all peaks on the carbon catalyst without modification, Ni-metal modification with 1, 2, and 3% have a fairly high intensity at $2\theta = 21.8; 21.9; 26.6; \text{ and } 26.5^\circ$, respectively. The diffraction pattern shows Nickel metal embedded into the activated charcoal catalyst. The highest conversion from catalytic cracking process with various Ni metal concentrations was 74.14% at temperature of 450 °C and concentration of 2% Ni-carbon. This conversion was higher than in the thermal cracking and catalytic cracking processes using a pure active catalyst. This means that there was an effect of nickel metal embedded in the pure activated carbon catalyst. From the density analysis, the liquid cracking process has in the range of 0.91772–0.95276 gr/ml, which is lower than crude palm oil as raw material. It indicated that long-chain hydrocarbons in crude palm oil breaks down and results in more carbon chains. When compared with the density of commercial liquid fuel, the density of the liquid resulting from this catalytic cracking is in the range of fuel oil and diesel.

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