Catalytic Cracking of Palm Fatty Acid Distillate with NaOH and KOH Catalyst Supported by Gamma Alumina

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Abstract. This article describe about the study of produce fuel from palm fatty acid distillate with a similar composition to fossil fuels through catalytic cracking method using alkaline heterogeneous catalyst. The catalytic cracking reaction was operated at batch reactor with a constant temperature of 370°C, a volume of 50 mL of feedstock, a pressure of 1 atm, and two kinds of catalysts: $NaOH/\gamma$-Al_2O_3 and KOH/γ-Al_2O_3 which has been characterization with X-ray diffraction and scanning electron microscopy. The best catalyst to produce biofuel type biogasoline (C_5-C_{15}) is KOH/γ-Al_2O_3 (5%) with a yield of 70% and selectivity to biogasoline of 74.46%. Meanwhile, the best catalyst to produce biofuel type biodiesel (C_{15}-C_{22}) is $NaOH/\gamma$-Al_2O_3 (5%) with a yield of 80% and selectivity to biodiesel of 67.72%.

Keywords: Biodiesel; Biogasoline; Catalytic Cracking; Palm fatty acid distillate

1. Introduction

The depletion of fossil fuels, coupled with the rapid growth in living standards, has led to a significant increase in fuel prices and the subsequent impact on greenhouse gas emissions. This has prompted researchers to prioritize the development of renewable and non-polluting fuels, which hold the promise of achieving global energy security while also mitigating the effects of climate change (Ibrahim, *et al.*, 2020; Singh *et al.*, 2020; Wakoc, *et al.*, 2018). Palm oils are widely considered to be the most promising feedstock for the production of such alternative fuels, known colloquially as biofuels. Nevertheless, if palm oil is used as a raw resource for biofuels for an extended period, it will eventually compete with foodstuffs. Using byproducts of palm oil processing, which still contain a lot of fatty acids as a raw material is a very wise decision (Oliveira, *et al.*, 2021; Arita *et al.*, 2020; Onlamnao and Tippayawong, 2020; Zaher, *et al.*, 2017).

Among all biofuel preparation technologies, catalytic cracking is considered a promising method due to its simple process which can be carried out at atmospheric pressure, has a higher cracking conversion efficiency, higher light alkene selectivity, less carbon deposition and the production of several types of liquid products similar to petroleum-based fuels. Furthermore, with appropriate catalysts, catalytic cracking can be used to produce gasoline, kerosene, and diesel, and it has good compatibility with various feedstocks as well as a lower cost (Ulfiati *et al.*, 2022; Zhang, *et al.*, 2021; Orazbayev, *et al.*,

2020; Nieuwelink *et al.,* 2020; Singh *et al.,* 2020; Mammadova *et al., 2018*). The cracking reaction is characterized primarily by a break in the "carbon-carbon" bond, indicating an endothermic reaction—the higher the temperature, the faster the reaction (Wang *et al.,* 2019). In comparison to the transesterification process, the catalytic cracking process is a more efficient method that offers greater flexibility in product output. The transesterification process, on the other hand, involves several complex steps, including the separation of products and by-products and the initial treatment of high FFA to prevent saponification reactions. Additionally, this process produces only one type of biofuel, and the production time can be relatively long, with processing times of around 5 hours and purification times of up to 24 hours (Wahyono *et al.,* 2022; Rasyid *et al.,* 2018). Meanwhile, when compared to the hydrocracking process, which requires high temperatures 350°C and 29.6077 atm pressures, the catalytic cracking process is more cost-effective because it can be operated at lower temperatures and atmospheric pressure (Trisunaryanti *et* al., 2020; Al-Muttaqi *et al.,* 2019; Rasyid *et al.,* 2015).

Currently, to meet the requirements of vehicle fuel standards, catalytic cracking must reduce the acid value and oxygen content of the biofuel which are biogasoline (C_5-C_{15}) dan biodiesel (C_{16}-C_{22}) to improve fuel properties such as density, calorific value, dynamic viscosity, and other parameters (Sardi *et al.,* 2022; Makertihartha *et al.,* 2020). There is no doubt that catalysts play an important role in the catalytic cracking of fatty acids to produce biofuel. Alkaline catalysts such as sodium hydroxide (NaOH) and potassium hydroxide (KOH) are commonly used in the production of biofuels because their rate of reaction is much faster than that of acid catalysts (Min *et al.,* 2015). High catalytic activity, shorter reaction time, simple operating conditions, low cost, and availability are just a few of the advantages of NaOH and KOH catalysts. However, homogeneous base catalysts, on the other hand, are extremely sensitive to free fatty acids and water. Therefore, a homogeneous catalyst was modified into a heterogeneous catalyst by the addition of a catalyst support (Widayat *et al.,* 2016).

Heterogeneous catalysts can be designed to graft and trap active molecules on the surface or within the pores of solid supports such as silica, alumina, or CaO (Thangaraj *et al.,* 2019). Gamma alumina (γ-Al_2O_3) is used as a catalyst support because it has a large surface area (150-300 m^2/g) and an amphoteric acid and base active site with various strengths depending on the method of production. Furthermore, γ-Al_2O_3 serves the primary function of providing surface area for the active component, with the purpose of increasing contact between the active core and the reactants while maintaining active phase activity (Rasyid *et al.,* 2018). The presence of acid sites on Al_2O_3, which are associated with Lewis acidity and very weak Brönsted acidity, makes it an effective catalyst for deoxygenation with minimal aromatization (Istadi *et al.,* 2021). Thus, making heterogeneous NaOH/γ-Al_2O_3 and KOH/γ-Al_2O_3 catalysts may be a feasible method to integrate the required acidic and basic characteristics to overcome defects in NaOH and KOH catalysts and avoid an excessive production of aromatic hydrocarbons by acid solid catalysts (Zheng *et al.,* 2019).

Therefore, the catalytic cracking of palm fatty acid distillate for biofuels was carried out using heterogeneous NaOH/γ-Al_2O_3 and KOH/γ-Al_2O_3 catalysts. Mechanically, NaOH/γ-Al_2O_3 and KOH/γ-Al_2O_3 catalysts were created by combining NaOH and KOH as primary catalysts and γ-Al_2O_3 as catalysts support. This research aims to produce a fuel with a similar composition to fossil fuels.

2.   Methods

2.1. Tools

The main tool in this research is a catalytic cracking reactor with supporting components (Figure 1).

Information :
a. Pressure gauge   h. Cooling water container
b. Thermocouple   i. Buffer
c. Inlet gas   j. Temperatur indicator
d. Valve   k. Heater button
e. Condensor   l. Power button
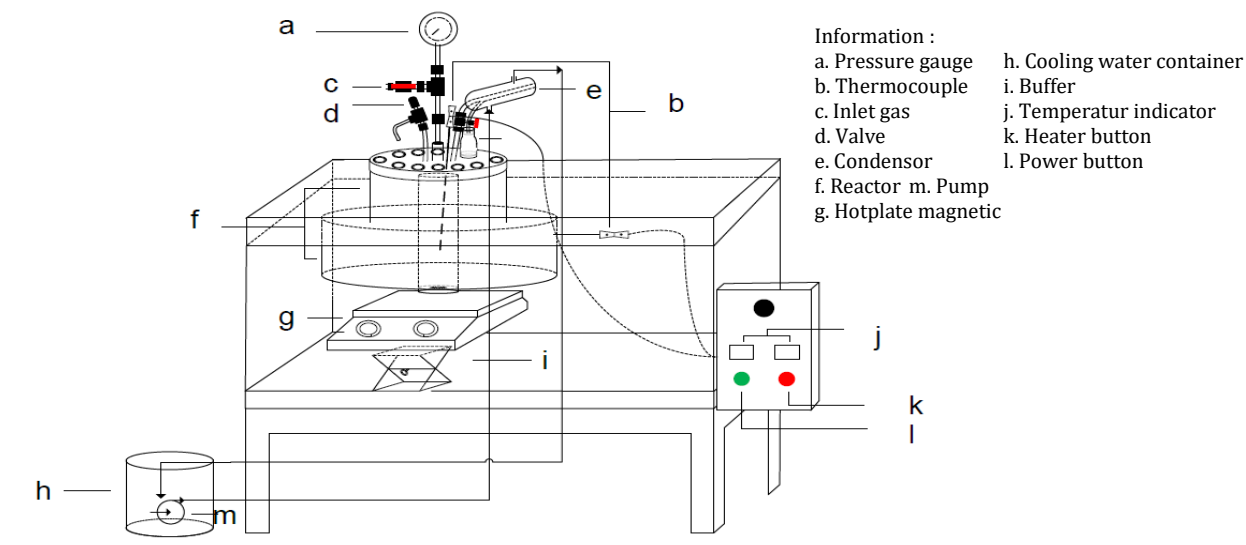f. Reactor m. Pump
g. Hotplate magnetic

Figure 1 Catalytic Cracking Reactor

2.2. Preparation of Catalysts

The catalyst was prepared from sodium hydroxide (NaOH) p.a merck and potassium hydorxide (KOH) p.a merck as catalyst site active element which impragnated into support gamma alumina (γ-Al$_2$O$_3$) from merck (p.a). Wet impregnation was used to prepare the catalysts. NaOH 0.5 N was impregnated into 10 grams of γ-Al$_2$O$_3$ to prepare NaOH/γ-Al$_2$O$_3$ catalyst, then it was stirred with a magnetic hotplate stirrer while the water in the mixture evaporated to form a paste. The mixture of the catalysts would be dried at 110°C within 8 hours. Afterward, the mixture was calcined for 3 hours at a temperature of 500°C. Furthermore, the same process was carried out with KOH 0.5 N to preparation KOH/γ-Al$_2$O$_3$ catalyst.

2.3. Catalytic Cracking Process

A batch reactor with a pressure of 1 atm was used to conduct the reaction. The reactor is filled with 50 mL of palm fatty acid distillate from palm oil refining, 0.5 grams of NaOH/γ-Al$_2$O$_3$ catalyst (1% of the raw ingredients), and a magnetic stirrer. The reaction is then performed for two hours after the reactor heater is turned on until it reaches a temperature of 370°C (Aziz *et al.,* 2020). The biofuel product will evaporate from the reactor to the liquid product container during the reaction and flow through the condenser. Remaining in the reactor is the residue, and the amount of gaseous product that hasn't condensed is estimated using the mass balance equation by deducting the initial amount of raw material from the final product. Furthermore, the catalytic cracking process is carried out with a NaOH/γ-Al$_2$O$_3$ catalyst (3, 5, 7)% and KOH/γ-Al$_2$O$_3$ catalyst (1, 3, 5, 7)%.

The yield product calculations and GC-MS analysis were carried out to determine the selectivity of the catalyst for biofuel products. The results of product yield and selectivity are obtained from equations (1) and (2).

$$\text{Yield} = \frac{\text{Product Volume (mL)}}{\text{Palm Oil Volume (mL)}} \text{ x } 100 \text{ \%} \qquad (1)$$

$$Selectivity = \frac{Selected\ product\ area}{The\ total\ area\ of\ all\ products}\ x\ 100\% \qquad (2)$$

2.4. Product Analysis

- Scanning Electron Microscopy (SEM) and X-Ray Diffraction (XRD) at the following conditions: 40 KV, 15 mA, CuK/1.54060 Time/step of 23.9700 s, step size of 0.0220 deg, and Scan axis Gonio were used to characterize the catalyst.
- Gas chromatography-mass spectrometry (GC-MS) with an Agilent capillary number of 19.091 S-493, HP-5MS of 5% phenyl methyl siloxane, nominal length of 30.0 m, nominal diameter of 250 um, nominal film thickness of 0.25 um, and nominal initial pressure of 10.5 psi was used to analyze the product's component compounds.

3.   Results and Discussion

3.1. Characterization of NaOH/γ-Al$_2$O$_3$ and KOH/γ-Al$_2$O$_3$ Catalyst

3.1.1 X-Ray Diffraction (XRD) Analysis

X-ray Diffraction (XRD) was used to identify content that was impregnated on the support of catalyst (γ-Al$_2$O$_3$) as the NaOH/γ-Al$_2$O$_3$ and KOH/γ-Al$_2$O$_3$ catalysts (Figure 2).
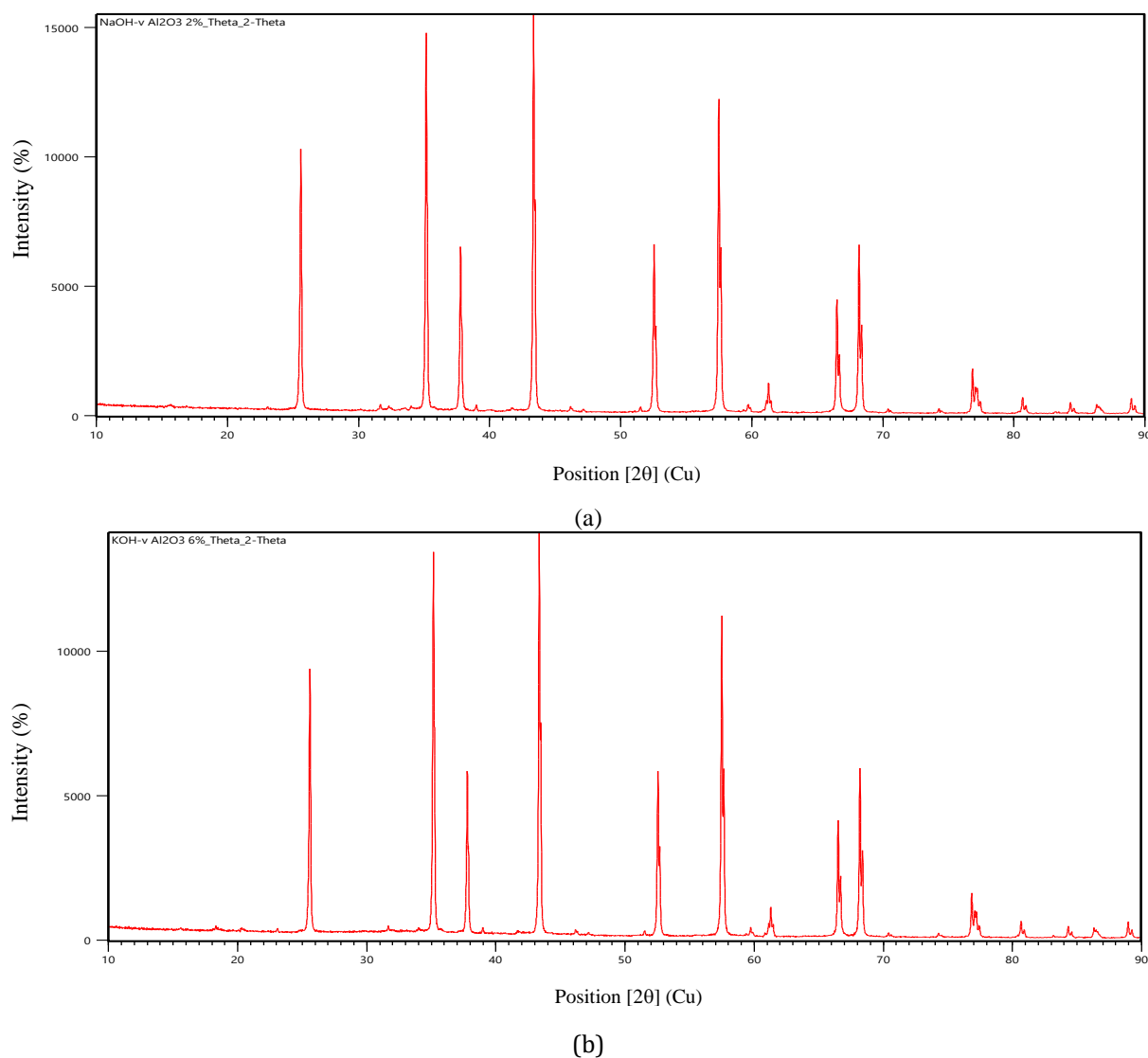

(a)

(b)

Figure 2 XRD diffractogram (a) NaOH/γ-Al$_2$O$_3$ catalyst, and (b) KOH/γ-Al$_2$O$_3$ catalysts

According to ICDD (International Center for Diffraction Data) 00-010-0425, Gamma Alumina (γ-Al$_2$O$_3$) has peaks at 2θ = 37°, 39°, 45°, and 67°. As shown in Figure 2, NaOH/γ-Al$_2$O$_3$ has peaks 2θ that are similar to γ-Al$_2$O$_3$, such as 37°, 39°, and 67°, but there are new peaks formed due to impregnation, indicating the presence of deposited NaOH catalyst. Likewise, the KOH/γ-Al$_2$O$_3$ catalyst has peaks 2θ that are similar to γ-Al$_2$O$_3$, notably 37°, 39°, and 67°, and there are new peaks are formed due to impregnation, proving the presence of deposited KOH catalyst (Singh *et al.*, 2020; Yu *et al.*, 2019; Wako *et al.*, 2018).

3.1.2 Scanning Electron Microscopy (SEM) Analysis

A SEM analysis was conducted to determine the surface morphology of the catalyst before and after impregnation of the active site of the catalyst. SEM analysis of the catalyst was carried out at a magnification of 10 μm (Figure 3).

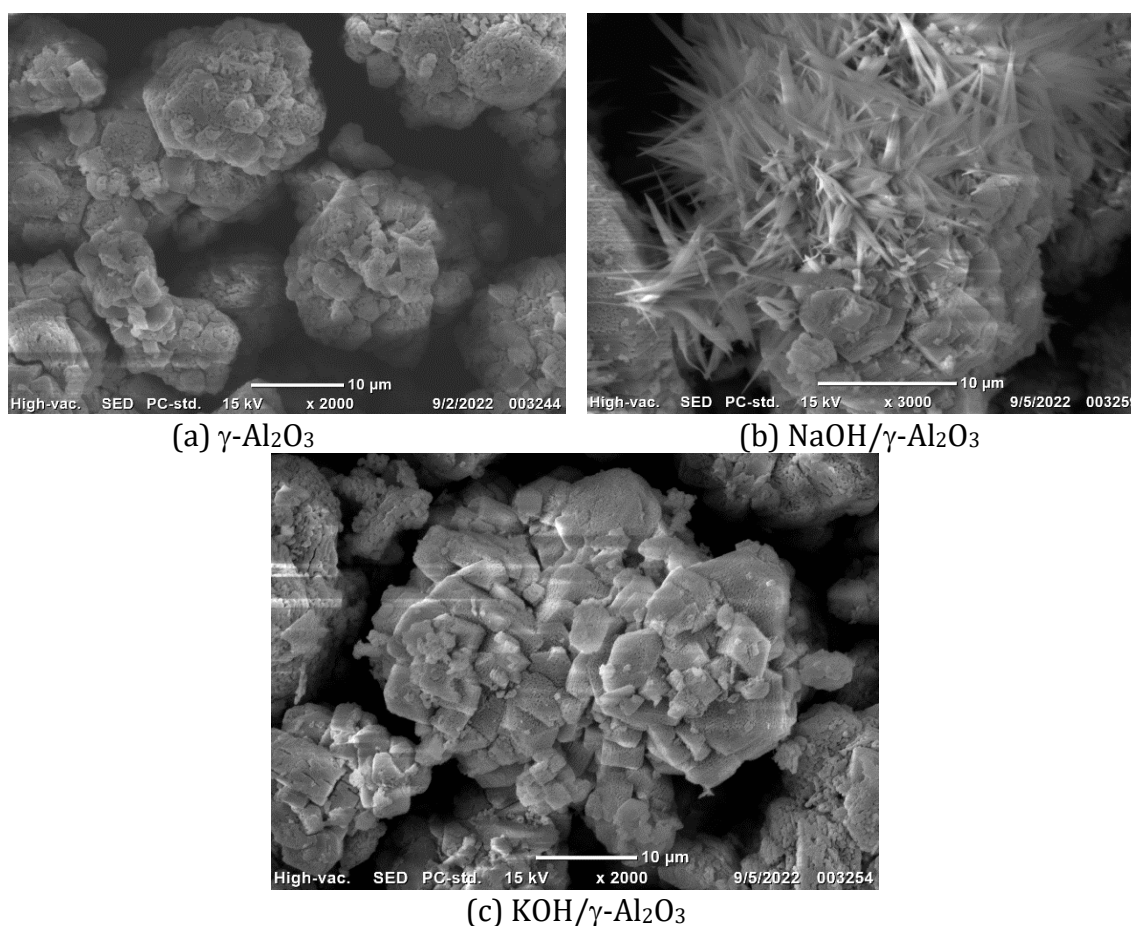(a) γ-Al$_2$O$_3$ (b) NaOH/γ-Al$_2$O$_3$

(c) KOH/γ-Al$_2$O$_3$

Figure 3 Surface Morfology of: (a) γ-Al$_2$O$_3$; (b) NaOH/γ-Al$_2$O$_3$; and (c) KOH/γ-Al$_2$O$_3$ catalysts

The surface morphology of the catalyst support ((γ-Al$_2$O$_3$) revealed a regular crystal structure before impregnation, whereas after impregnation revealed that the impregnated active site had attached and distributed to the support's main structure.

3.2. Gas Chromatography-Mass Spectrometry Analysis of Palm Fatty Acid Distillate

Palm fatty acid distillate was GC-MS analyzed before the catalytic cracking process to determine the compound composition of the raw material used (Table 1). The by-product of palm oil processing used in this study still contains triglyceride components, specifically free fatty acids. As noted by (Oliveira *et al.*, 2021), the conversion of palm oil into cooking oil can result in up to 6% of the by-product of feed CPO being left behind. The byproduct is palm oil fatty acid distillate, which contains a high concentration of free fatty acids. There

are also significant amounts of hydrocarbon compounds (Table 1). This shows that palm oil waste has the potential to be used as a raw material in the production of biofuels.

Table 1 The Compound Composition of Palm Fatty Acid Distillate

Chemical Formula	Composition(%)	Chemical Formula	Composition(%)
$C_3H_4N_2O$	0.27	$C_{11}H_{22}O_2$	0.39
$C_3H_6N_4O_3$	0.35	$C_{11}H_{23}O_2$	1.60
$C_5H_8O_2$	0.24	$C_{11}H_{24}$	1.07
$C_5H_{10}O$	0.63	$C_{11}H_{10}$	1.86
C_6H_{12}	2.35	$C_{12}H_{26}$	14.77
$C_6H_{10}O_2$	1.73	$C_{12}H_{24}$	4.15
C_7H_{16}	2.84	$C_{12}H_{18}O$	0.93
C_7H_{10}	0.30	$C_{12}H_{26}O$	0.31
$C_7H_{10}O_2$	2.26	$C_{13}H_{28}$	12.99
$C_7H_{16}O$	4.22	$C_{14}H_{28}$	2.58
C_8H_{18}	0.64	$C_{15}H_{30}$	2.4
C_8H_{16}	2.11	$C_{16}H_{30}$	1.17
$C_8H_{15}N_3O$	1.39	$C_{18}H_{34}O_2$	1.01
$C_9H_{18}O$	0.83	$C_{18}H_{36}O_2$	6.38
C_9H_{16}	1.96	$C_{19}H_{30}O$	1.99
$C_{10}H_8$	2.66	$C_{20}H_{20}O_4$	1.78
$C_{10}H_{16}O$	0.2	$C_{20}H_{36}O_2$	2.08
$C_{10}H_{18}O$	0.79	$C_{26}H_{34}O_{10}$	1.19
$C_{10}H_{22}$	0.28	$C_{28}H_{34}N_2O$	1.13
$C_{10}H_{22}$	9.48	$C_{36}H_{74}$	2.84
$C_{10}H_{14}O_2$	0.23	CH_4N_2O	0.77

3.3. Product Yield Analysis Results

Biofuel products are obtained from the catalytic cracking process in the form of liquid and gas, and also residues, which are raw materials that are not cracked. The liquid and gas results are added up and then calculated using equation (1) to determine the yield of each reaction.
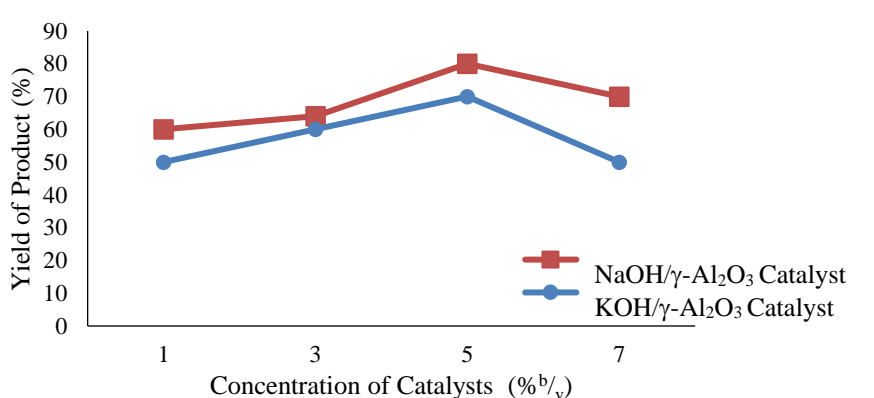

Figure 4 Graph of the Relationship between Concentration of Catalysts and Yield Product

The yield of the product obtained in the biofuel production process by catalytic cracking of palm fatty acid distillate with NaOH/γ-Al$_2$O$_3$ and KOH/γ-Al$_2$O$_3$ catalysts was directly proportional to the catalyst concentration up to 5% and experienced product yields at 7% catalyst concentration (Figure 4). Studies by (Thambiyapillaia and Ramanujam, 2021; Akah, Williams, and Ghrami, 2019; Prabasari, *et al.*, 2019) have demonstrated that the addition of a catalyst to a catalytic cracking reaction can increase the reaction rate, resulting in higher yields. However, it is important to note that if the catalyst's performance has already reached its optimum limit, adding more of it beyond that point will not lead to further improvements in the reaction's effectiveness

Despite the similarities in the line chart, the amount of yield produced by each catalyst is different. The yield obtained with the NaOH/γ-Al$_2$O$_3$ catalyst was greater than the yield obtained with the KOH/γ-Al$_2$O$_3$ catalyst. The product yield obtained with the NaOH/γ-Al$_2$O$_3$ (5%) catalyst was 80% while the higher yield of KOH/γ-Al$_2$O$_3$ (5%) was 70%. This is due to the active site distribution of the NaOH/γ-Al$_2$O$_3$ catalyst obtained through scanning electron microscopy analysis appearing wider and adhering more to the catalyst support (Figure 3).

3.3. Selectivity Product

The biofuel product produced through the cracking process of palm fatty acid distillate using a NaOH/γ-Al$_2$O$_3$ catalyst and a KOH/γ-Al$_2$O$_3$ catalyst contains various hydrocarbon compounds, so gas chromatography-mass spectrometry analysis is carried out. Biofuels are classified based on their constituent hydrocarbon chains, which are biogasoline (C$_5$-C$_{15}$) dan biodiesel (C$_{16}$-C$_{22}$) (Aziz *et al.*, 2021a ; Onlamnao and Tippayawong, 2020; Ibarra *et al.,* 2019). Then calculate the selectivity of the catalyst for the type of biofuel produced by using equation (2).
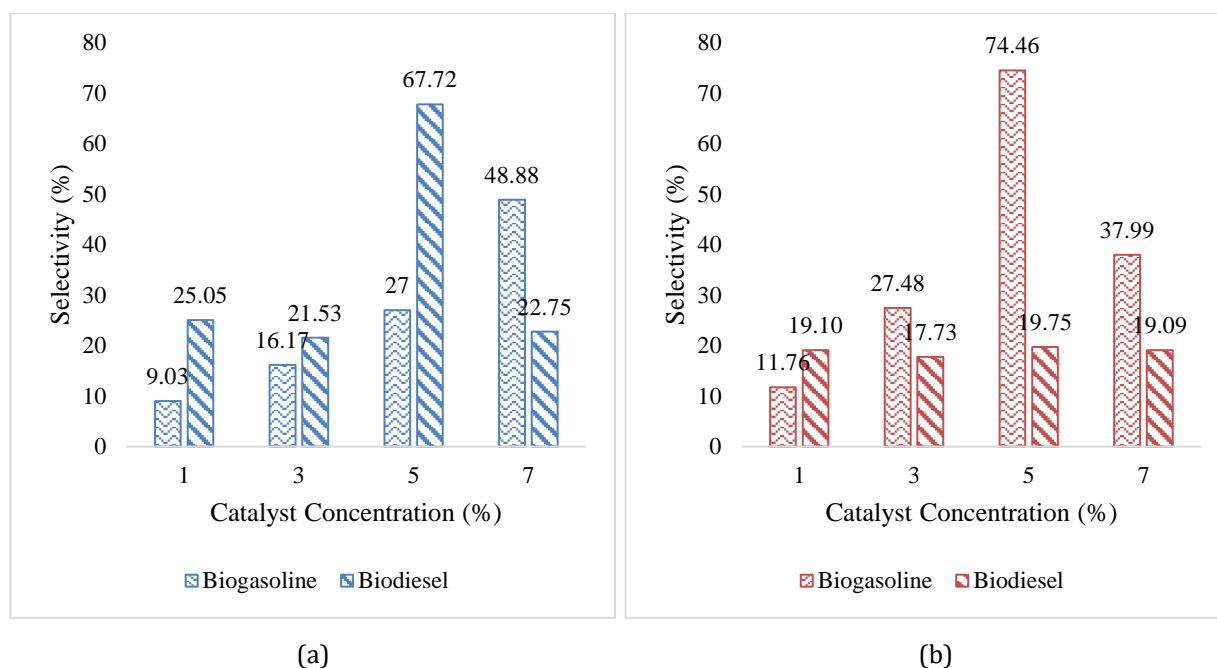

(a)   (b)

Figure 5 Graph of Relationship between Catalyst Mass and Selectivity of Biofuel Products using a catalyst (a) NaOH/γ-Al$_2$O$_3$; and (b) KOH/γ-Al$_2$O$_3$ catalysts

The highest conversion of biodiesel product from the catalytic cracking of palm fatty acid distillate was obtained with NaOH/γ-Al$_2$O$_3$ (5%) catalyst, which was 67.72%. This shows that the NaOH/-Al$_2$O$_3$ catalyst is more selective towards long-chain biofuels (C$_{15}$-C$_{22}$) compared to the KOH/γ-Al$_2$O$_3$ catalyst, which produces less than 20% biodiesel.

Meanwhile, the conversion of biogasoline products from the catalytic cracking of palm fatty acid distillates with the NaOH/γ-Al$_2$O$_3$ catalyst shows data that is directly proportional to the increase in the catalyst. The highest biogasoline product with NaOH/γ-Al$_2$O$_3$ catalyst was obtained at 7% catalyst, which was 48.88%, indicating that if the catalyst is increased again, the conversion may increase or decrease. However, when compared to the catalytic cracking of palm fatty acid distillate with a KOH/γ-Al$_2$O$_3$ catalyst, better results were obtained, where the optimum biogasoline production point was obtained with a KOH/γ-Al$_2$O$_3$ catalyst (5%) and a bio gasoline yield of 74.46%. This shows that the KOH/γ-Al$_2$O$_3$ catalyst is more selective towards short-chain biofuels (C$_5$-C$_{15}$) than the NaOH/γ-Al$_2$O$_3$

catalyst. According to (Aziz *et al.*, 2021b; Istadi *et al.*, 2021; Senter *et al.*, 2021), catalysts that produce short-chain biofuels have high performance. The product's selectivity is proportional to the Lewis to Brønsted ratio (L/B ratio). When the L/B ratio is high, the Lewis acid site is dominant. Because of the catalyst's low L/B ratio, the NaOH/γ-Al$_2$O$_3$ catalyst promotes the formation of long chains (biodiesel).

4. Conclusions

Palm fatty acid distillate (PFAD) can be converted into biogasoline (C5–C15) and biodiesel (C16–C22) at a pressure of 1 atm using NaOH/γ-Al$_2$O$_3$ and KOH/γ-Al$_2$O$_3$ as catalysts in a catalytic cracking process. KOH/γ-Al$_2$O$_3$ (5%) is the best catalyst for producing biofuel type biogasoline (C5-C15), with a yield of 70% and a selectivity to biogasoline of 74.46%. Meanwhile, the best catalyst for producing biofuel type biodiesel (C15-C22) is NaOH/γ-Al$_2$O$_3$ (5%), which has an 80% yield and a 67.72% selectivity for biodiesel. Furthermore, the product of this research can be utilized as a blend of commercial fuels, given that they contain the same compounds and that the combustion products are easily decomposed, minimizing pollution to the environment. It is also intended to reduce the use of fossil fuels, ensuring global energy availability. This research can be expanded with different pre-treatments in the manufacture of catalysts to increase their effectiveness, as well as the addition of appropriate promoters.

Acknowledgments

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