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Synthesizing and Performance Testing of Zn Promoted Ni Catalyst With γ-Al_2O_3 Support in The Process of Hydrotreating Used Cooking Oil into Green Diesel

Faizal Zul Kopli[1], Fadel Kurnia Artha[1], Ismeini[1], Erlinawati*[1], Adityas Agung Ramandani[2], Dimas Amirul Mukminin Nur Efendi[3]

[1]Departement of Chemical Engineering, Sriwijaya State Polytechnic, Indonesia
[2]Departement of Chemical Engineering and Materials Science, Yuan Ze University, Taiwan
[3]Departement of Chemical Engineering, Lampung State Polytechnic, Indonesia

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ABSTRACT

Green diesel was a mixture of straight-chain and branched-chain alkanes, typically mostly 15 to 18 carbon atoms per molecule (C15 to C18) extracted from the hydrotreating process of vegetable oils. In this study, a catalyst of Nickel (Ni) promoted Zinc (Zn) supported by Alumina (Al_2O_3) was used. The catalyst was made by varying the use of and without the Zn promoter in the catalyst. Catalyst tested for Scanning Electron Microscope characterization. For the $60Ni/Al_2O_3$ catalyst, the total area was 51,575.51 m2/gr, while for the $15Ni\text{-}45Zn/Al_2O_3$ catalyst, the total area was 20.577,55 m2/gr. Furthermore, a performance test of the catalyst was also carried out using a green diesel reactor with a temperature of 400°C and a pressure of 20 bar, the percentage yield on the $60Ni/Al_2O_3$ catalyst was 25.73% while the $30Ni\text{-}30Zn/Al_2O_3$ catalyst was 23.60%. The $60Ni/Al_2O_3$ catalyst has the following properties: physical properties such as density, viscosity, flash point, cetane number, and acid number of 765.17 ± 0.249 kg/m^3, 3.55 ± 0.076 mm^2/s, 60.73 ± 0.170°C, 88.10 ± 0.648 CN, and 0.33 ± 0.76 mgKOH/g, respectively. The $15Ni\text{-}45Zn/Al_2O_3$ catalyst has the following properties: physical properties such as density of 766.77 ± 0.679 kg/m^3, viscosity of 66.13 ± 0.249 mm^2/s, flash point of 2.92 ± 0.011°C, cetane number of 82.33 ± 0.386, and acid number of 82.33 ± 0.386 mgKOH/g.

1. INTRODUCTION

The depletion of natural resources, particularly petroleum, has left Indonesia with complicated energy sector issues. A previous study conducted by Outlook (2019) has reported that the decrease in petroleum output is stated in the Ministry of Energy and Mineral Resources. From 2009 to 2018, the amount produced decreased from 346 million barrels to 283 million barrels. The D100 program, which is a vegetable oil-based diesel fuel without blending with petroleum-based diesel, including green diesel, is being promoted by the government (Doğan & Erol, 2023; Malabadi et al., 2023). Vegetable oil, such as discarded cooking oil, is widely available in Indonesia (Apriantoro et al., 2023; Dewi & Iskandar, 2023; Febijanto et al., 2023). In addition, wasted cooking oil still has the potential to be turned into fuel because it still has the same properties as other vegetable oils, specifically the presence of fatty acids and triglycerides. A recent study conducted by Mannu et al. (2019) provides evidence for this claim, demonstrating that used cooking oil comprises 70% triglycerides as oleic acid, 13% as linoleic acid, and less than 3% as linolenic acid.

A recent study conducted by Zhang et al. (2019) used cooking oil as fuel. In this study, spent cooking oil was hydrogenated catalytically using the MCM-41 zeolite catalyst, which resulted in the production of aviation fuel and paraffin fractions at 400°C to 420°C and pressures as high as 37% at 10 Bar (C10-C14). The study found that the aviation fuel

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*Correspondence author.
E-mail: erlinawatiakil@yahoo.com (Erlinawati)

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produced had a high cetane number and low sulfur content, making it a promising alternative fuel source (Mofijur et al., 2023; Undavalli et al., 2023). However, green diesel has not been the result of this research (C15–C18). This results from the choice of an inappropriate catalyst, which is to be expected given that transition metals are resistant to high working temperatures. This is because the reaction that was employed was an exothermic hydrogenation process (van Dyk et al., 2019). Additionally, transition metals have greater activity in hydrogenation processes, where they break double bonds in the fatty acids found in raw materials. This is so that they can form bonds with hydrogen atoms that will be introduced throughout the reaction process, which is why transition metals have vacant d orbitals (Wu et al., 2023; Zhang et al., 2023). Therefore, used cooking oil was hydrotreated using a Ni-Mo/γ-Al_2O_3 catalyst (Nugraha et al., 2021). The results showed a significant increase in the yield of biodiesel. In this study, the highest percentage of green diesel yield 35.80% was attained after five hours at 430°C and an ideal pressure of 4 bar. In a different study involving transition metal catalysts, vegetable oil was hydrotreated using a Ni-Zn/γ-Al_2O_3 catalyst, which was able to convert 99% and produce 71.96% of green diesel in a semi-batch reactor operating at 310°C and 40barr of pressure. Based on the research conducted by Gousi et al. (2017) has reported the use of Ni/γ-Al_2O_3 as a transition metal catalyst in the hydrotreating of vegetable oil. The process can yield 61% green diesel when conducted in a semi-batch reactor at 310°C and 40 bar of pressure, with 60% Ni and 40% γ-Al_2O_3 as the catalyst composition. This study demonstrates that the hydrotreating method can potentially improve the selectivity of green diesel products by utilizing transition metals, such as nickel, supported by aluminum supports.

Consequently, further study is required to create Ni/γ-Al_2O_3 transition metal catalysts that include a zinc promoter to hydrotreat used cooking oil and turn it into green diesel. This could potentially lead to a more efficient and sustainable method for producing biofuels. Following that, a quality test of the generated green diesel product was conducted, along with a catalyst characterization test and a catalyst performance test on the product's selectivity.

2. METHODS

The research was carried out over four months, starting from April to July 2022, and was carried out at the Energy Engineering Laboratory and the Chemical Engineering Laboratory of the Sriwijaya State Polytechnic. Materials that have been used in this research include cooking oil, nickel (II) nitrate (Ni $(NO_3)_2$).$6H_2O$), and zinc nitrate (Zn $(NO_3)_2$). $6H_2O$), aluminum nitrate ((Al $(NO_3)_3$).$9H_2O$), NH_4OH 25%, aquadest, hydrogen gas, aluminum foil, and filter paper (size). Apart from that, the tools used in this research were a furnace, spatula, 25 mL measuring pipette, 50 mL measuring cup, 1 L Erlenmeyer, 500 mL separating funnel, vacuum pump, green diesel reactor, 5 mL pycnometer, and viscometer.

2.1 Catalyst synthesis

The methods used in this study are inspired by and differ from those described in (Gousi et al., 2017). In an Erlenmeyer flask, dissolve Ni $(NO_3)_2.6H_2O$, Zn $(NO_3)_2.6HO$, and Al $(NO_3)_3.9H_2O$ simultaneously with different catalyst compositions (Table 1), stirring continuously for 24 hours. The sample solution should be placed in a glass funnel and gradually poured into a beaker holding a 25% NH_4OH solution. After ten minutes from the initial drop, the four-hour deposition procedure starts. Utilizing a vacuum pump, filter the resultant suspension. produces solids with a size of less than 10 mm. After the solid has been dried for 24 hours at 110°C, it is calcined for 2.5 hours at 400°C. The catalyst is subjected to SEM examination after printing to ascertain its overall area, pore count, and average pore size. At LTJIST, SEM testing was done by (Ramandani et al., 2022).

Table 1. Experimental Design for Synthesis Catalyst

Combination	Ratio catalyst (%)		
	Ni	Zn	Al_2O_3
1	60	40	0
2	45	15	40
3	30	30	40
4	15	45	40
5	5	55	40

2.2 Application of catalyst to green diesel using used cooking oil

Once the catalyst has been created, it is used in the green diesel reactor to assess how effective it is. Reactor I should receive two liters of used cooking oil (R1). Fill Reactor III (R3) with 100 grams of catalyst. Shut R1 and R3, then tighten the cover bolts. Put gas containing hydrogen into the accumulator tank. Set the temperature to 400°C and turn on the band heaters R1 and R3. Two hours are dedicated to the heating process. Subsequently, the separator tank can be used to extract the product sample. After every data collection cycle, turn it off and let the hydrogenation reactor cool. quantifying the amount of the acquired results. Conduct quality testing on each generated product by measuring its cetane number (ASTM D975), acid (ASTM D664-04) density

(ASTM D976), viscosity (ASTM D445), and flash point (ASTM D93).

Figure 1. Green diesel reactor-based cooking oil using catalyst products.

2.3 Examination of product yields

To determine the catalyst's percent yield, the catalyst's performance was evaluated during the hydrotreating step in the production of green diesel in a green diesel reactor. Two liters of spent cooking oil were utilized as the raw material, and the reactor was operated at 400°C and 20 bar of pressure. To calculate the yield product, as shown in Equation 1.

$$Yield\ (\%) = \frac{A}{B} \times 100 \tag{1}$$

Where, A and B are the mass of green diesel and the mass of raw materials, respectively.

2.4 Green Diesel Density Test (D4737 / D976 ASTM)

Raise the temperature of the distilled water to 40°C. Weigh the dry and empty pycnometer. After 40°C, fill the pycnometer with pure water, whose density is reported in the literature to be 0.99 kg/m^3. After heating the green diesel product to 40°C, pour it into the pycnometer. Use equation 2 to determine the density of green diesel at 40°C.

$$\rho = \frac{m}{v} \tag{2}$$

Where, ρ is density, m is the mass of green diesel, and v is the volume of green diesel.

2.5 Green Diesel Kinematic Viscosity Test (ASTM D445)

Heating the sample of green diesel to 40°C. After measuring the ball's diameter, weigh it with an analytical balance. To calculate density and volume, as shown in Equation 2 and 3, respectively. The ball should be placed on the viscometer's surface, and released, and the distance it takes the ball to travel 20 cm should be timed. After three repetitions, use Equation 4 to determine the average ball travel time. Apply Equation 5 to determine the dynamic viscosity of green diesel.

$$V = \frac{4}{3}\pi\left(\frac{d}{2}\right)^3 \tag{3}$$

$$t = \frac{t_1 + t_2 + t_3}{3} \tag{4}$$

$$\mu = K \times (\rho_1 - \rho_2) \times t \tag{5}$$

Where, t is the time, μ is the viscosity, ϱ is the density, K is constant (0.09) and V is the volume.

2.6 Green Diesel Flash Point Test (ASTM D93)

Before measuring the flash point, clean the flash point tester. Once the green diesel sample has reached its limit, fill the metal vessel, and shut it. Put the thermometer and stirrer in place. Connect the burner gas pipe and install the existing connection cable. Switch on the electricity and gas hobs. Adjust the heating so that the temperature rises by 5 degrees Celsius every minute, then watch until the substance's vapor burns completely, and the temperature is noted. Turn off the Flash Point Tester after using it for the last time, then clean it one more.

2.7 Green Diesel Cetane Number Test (ASTM D975)

Press the on button located on the electrical panel to switch on the electricity. Next, turn on the chimney and socket. Turn on the cooling water valve and flip the engine panel's start position on the CFR. Activate the temperature controller and air heater by turning them on. Set the position of the lubricant temperature control. After the engine has warmed up for 30 to 45 minutes or until the lubricant temperature reaches 220°F, stop. This machine may be heated using fuel or electricity. Measure the 60 ml/minute (60 ± 1 second per 13.0 ml) fuel flow rate to the pump or injection flow rate. Pour test fuel into the second tank. Using the fuel injection angle panel as a guide, set the fuel injection timing to 13°. Turn the handwheel until the fuel injection angle panel indicates an ignition delay at position 13°. Take note of the handwheel's scale. In tank number one, enter the comparison fuel, whose cetane number is known. Refresh the fuel pump. As in the example, adjust the fuel injection time and ignition delay. Take note of the reading on the handwheel. In tank number three, enter comparison fuel II, whose cetane number is known. The sample's cetane number and the cetane numbers of the comparative fuels I and II must be between each other, with a maximum difference of 5 cetane numbers between them. adjusting the ignition delay and injection time. On the handwheel scale, note the final reading.

3. RESULT AND DISCUSSION

3.1 Characteristics of catalyst products

The synthesis approach yielded a nickel (Ni) catalyst with γ-Al_2O_3 support, both with and without the addition of a zinc promoter. In the hydrotreating process, catalysts are compounds that are used to speed up the chemical reactions that turn spent cooking oil and hydrogen gas into hydrocarbons. With a fraction range of C15–C18, green diesel is the desired product of this procedure. Heterogeneous catalysts include Ni catalysts both with and without the inclusion of a Zn promoter. Specifically, a catalyst that exists in a phase distinct from the reactants. The catalyst that is employed is a solid phase. Cooking oil that has been utilized and hydrogen as reactants are currently in the liquid and gas phases. An extensive application of heterogeneous catalysts is in surface reactions like adsorption. Due to their resistance to phase transitions after reactions, transition metals are employed as catalysts. Although catalysts don't supply energy to the system, they do offer a reaction mechanism whose activation energy is lower than in reactions that don't involve one, which causes the reaction rate to increase when a catalyst is present.

Table 2. Mass of catalyst products

Catalyst composition	Mass of catalyst (grams)
60% Ni/40% Al_2O_3	104,66
45% Ni-15% Zn /40% Al_2O_3	102,65
30% Ni-30% Zn /40% Al_2O_3	101,94
15% Ni-45% Zn /40% Al_2O_3	106,68
5% Ni-55% Zn /40% Al_2O_3	101,36

In the formulation without the zinc promoter, 104.66 grams of catalyst were effectively synthesized. Meanwhile, 106.68 grams were achieved using a Zn promoter that was 15% Ni-45% γ-Al_2O_3. The weights of 131.416 grams and 135.197 grams, which result from theoretical stoichiometry calculations, differ significantly. There are multiple reasons why this occurs. The initial step involves moving the catalytic processing from one container to another. Second, too much ammonia is causing a ligand exchange reaction. A metal amine complex is created when the excess ammonia in the air prevents metal hydroxide from forming (Apriantoro et al., 2023).

3.2 The outcomes of the catalyst characterization test.

The shape and surface structure of the catalyst was examined using a scanning electron microscope (SEM) at 20,000x magnification as part of the catalyst morphology characterization test. Table 2 provides evidence of it. Images are formed by electrons in this characterization test, which is conducted under vacuum conditions. Using ImageJ software, tests were conducted to ascertain the catalyst sample's particle size. Before calibrating the software, the picture from the SEM test results is used to determine the image type, which is set to 8-bit with a scale size of 1μm. Then, make the visual effect darker or lighter so that it is easier to observe the surface and analyze the particle size. After that, click the Analyse menu> Analyse Particle> Outlines. From the results of the data analysis, the number of pores, total area, and average pores were obtained.

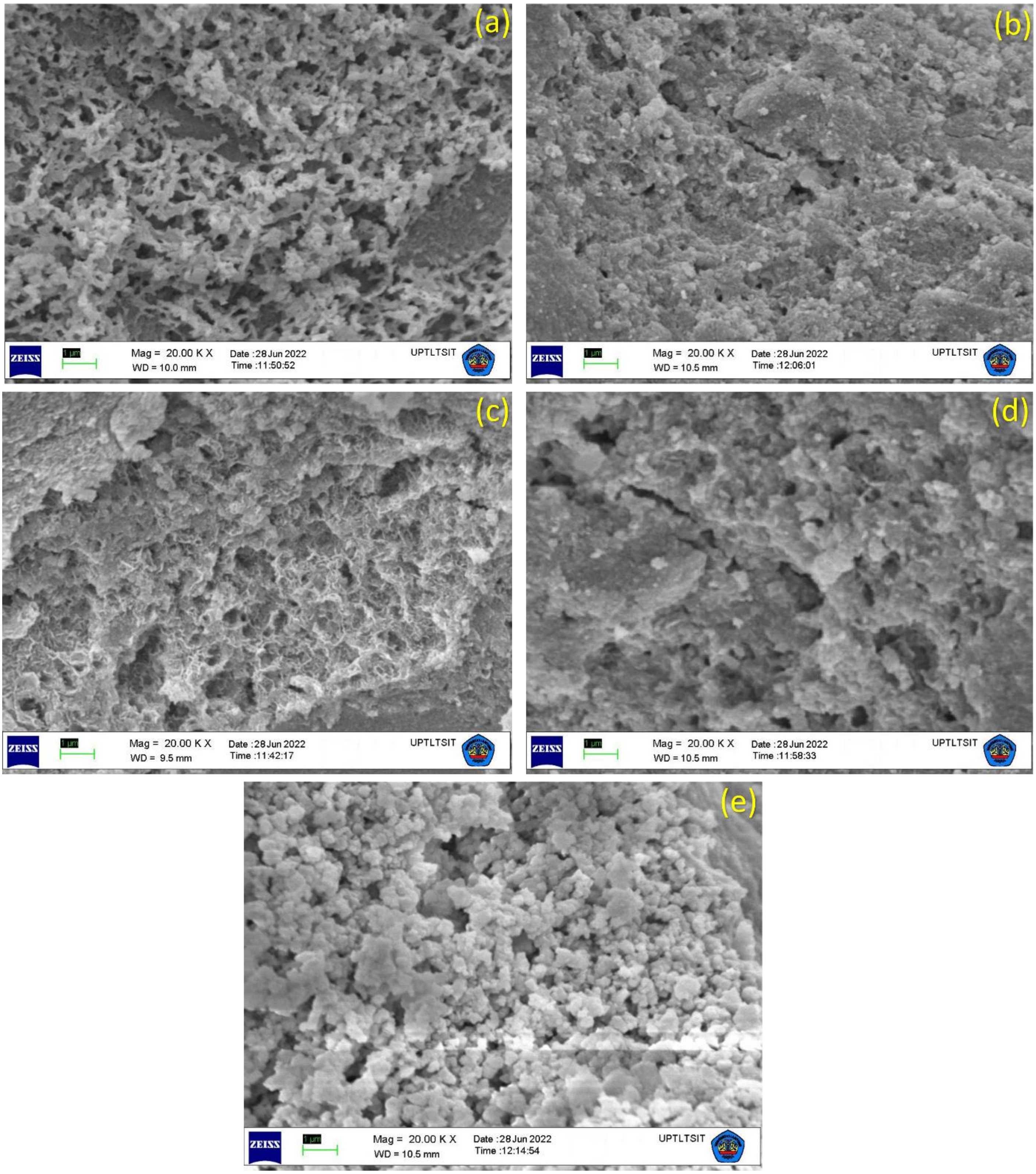

Figure 2. Scanning Electron Microscope with various catalyst compositions of (a) 60%Ni/ 40% Al_2O_3, (b) 30%Ni- 30% Zn/ 40% Al_2O_3, (c) 45%Ni- 15% Zn/ 40% Al_2O_3, (d) 30%Ni- 30% Zn/ 40% Al_2O_3, and (e) 5%Ni- 55% Zn/ 40% Al_2O_3

Table 3. Number of pore catalysts, size total, and average size-based SEM analysis

Catalyst composition	Number of pores (µm)	Size total (µm²/gr)	Average size (µm²)
60% Ni/40% Al_2O_3	684	51,575.511	75.403
45% Ni-15% Zn /40% Al_2O_3	363	26,126.531	71.973
30% Ni-30% Zn /40% Al_2O_3	523	20,979.592	40.114
15% Ni-45% Zn /40% Al_2O_3	579	20,577.551	35.539
5% Ni-55% Zn /40% Al_2O_3	134	3,118.367	23.271

In comparison to 15Ni-45Zn/γ-Al_2O_3, the 60Ni/γ-Al_2O_3 catalyst has more pores overall and in comparison, to the data in Table 3. The catalyst's inclusion of the Zn promoter is the reason for this. The catalytic core is shielded by the promoter itself. On the other hand, too much promoter may mask some of the catalyst and support (Apriantoro et al., 2023). The zinc promoter requires a wide surface area, which makes it excellent for reactions involving hydrogen. It is also easily shaped, has a high melting point, and is relatively stable at high temperatures (Hooshmand et al., 2023; Rahim et al., 2023).

3.3 Catalyst Performance Test

To find the yield percentage on green diesel products, performance tests are conducted. The impact of the 60Ni/γ-Al_2O_3 catalyst, both with and without a Zn promoter added to the yield obtained from hydrotreating used cooking oil was investigated. The amount of product generated from the amount of raw material fed is expressed as a yield percentage. The amount of material that is effectively transformed into a product relative to the amount of feed material is known as the percent yield; the more product produced, the higher the percent yield. The mass of the green diesel product divided by the quantity of used cooking oil fed is the computed yield percentage. Figure 3 below shows the graph of the 60Ni/γ-Al2O3 catalyst with and without the Zn promoter.

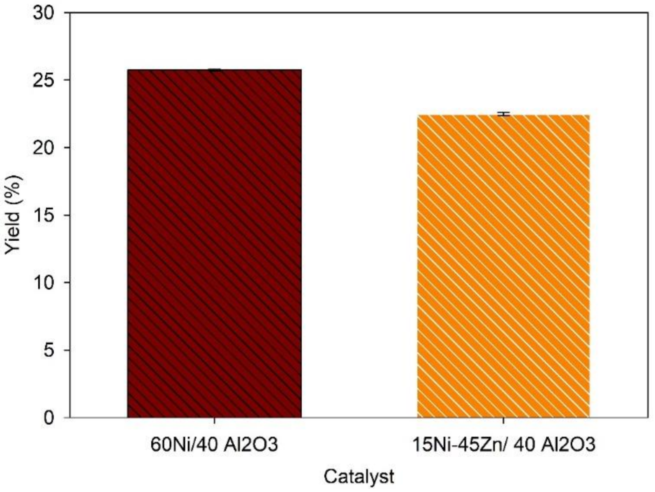

Figure 3. Performance test results for green diesel products using two catalysts with higher pore size

Figure 3 displayed the product yield generated by catalysts 15Ni-45Zn/γ-Al_2O_3 and 60Ni/γ-Al_2O_3. The catalyst 60Ni/γ- Al_2O_3 displayed a yield of 25.73%. This yield is higher than that of 15Ni-45Zn/γ- Al_2O_3, which yields 22.48%. This suggests that the amount of product that will be generated will increase with the proportion of nickel content. The catalyst's huge reactive surface area explains this. Aside from that, according to Rasyid (2022), adding a metal promoter to the support will fill its pores and decrease its current surface area. In this case, Zn is the promoter metal that has been added. This metal fills the Al_2O_3 pores, decreasing the catalyst's overall surface area. This influences the yield outcomes obtained as a percentage.

3.4 Characteristics of green diesel

Table 4 displays the property values of the manufactured green diesel product. The range of densities for green diesel depends on the norms that are applied. 765–800 kg/m³. Upon examining the density requirements of individual products derived from each catalyst, it is evident that the 60Ni/γ-Al_2O_3 catalyst yielded lower density results 765.2 against 45Zn/γ-Al_2O_3. However, the density difference between the two catalysts was not significant enough to impact the overall performance of green diesel. The selectivity performance of the catalyst is reduced when an excessive amount of Zn promoter is added (Gousi et al., 2020). Therefore, the lower density findings found can potentially be attributed to a decrease in product selectivity caused by the presence of a Zn promoter in the 60Ni/γ-Al_2O_3 catalyst. Thus, it is essential to keep the Zn promoter level at its ideal level to achieve the required product density and engine performance.

Table 4. Characteristics of green diesel products based on two catalysts, such as 60% Ni/40% Al_2O_3 and 15% Ni 45% Zn /40% Al_2O_3

Parameter	**Standard**	**Catalyst**	
		60% Ni/40% Al_2O_3	**15% Ni 45% Zn /40% Al_2O_3**
Density (kg/m^3)	765-800	765.17 ± 0.249	766.77 ± 0.679
Viscosity (mm^2/s)	2 – 4,5	3.55 ± 0.076	2.92 ± 0.011
Flashpoint (°C)	Min 55	60.73 ± 0.170	66.13 ± 0.249
Cetane Number (CN)	70-90	88.10 ± 0.648	82.33 ± 0.386
Acid number (mg KOH/gr)	>0,1	0,33 ± 0.76	0.08 ± 0.011

Source: **Green Diesel European Standard EN15940:2016/A1:2018*

Aside from that, according to the Green Diesel European Standard EN15940:2016/A1:2018, the viscosity of green diesel fuel varies from 2.0 to 4.5 mm^2/s. Based on study data, the viscosity value for the product generated by the 60Ni/γ-Al_2O_3 catalyst was found to be 3.55 ± 0.076 mm^2/s. This viscosity rating for green diesel fuel is within the typical range. On the other hand, 2.92 ± 0.011 mm^2/s was the viscosity value obtained employing the 15Ni-45Zn/γ-Al_2O_3 catalyst. Lower viscosity values are seen in the catalyst 15Ni-45Zn/γ-Al_2O_3. There is a greater viscosity value in the 60Ni/γ-Al_2O_3 catalyst. This is due to the presence of heavy fractions in the C15–C18 range of green diesel, which causes a higher viscosity. This is consistent with the view expressed by Othman et al. (2017) and Asikin-Mijan et al. (2017) this study claims that the hydrocarbon chains created during cracking have an impact on the viscosity of the resulting green diesel fuel. The study suggests that the choice of catalyst can significantly influence the viscosity of green diesel fuel produced through catalytic cracking processes. In addition, this is because an increase in viscosity corresponds to a closer intermolecular density, which in turn increases the cohesive force inside the oil and raises its viscosity (Heris et al., 2024; Sulaimon & Adeyemi, 2018). Low viscosity facilitates better combustion by facilitating the injection and mixing of fuel with air (Hamid et al., 2020; Hwang et al., 2017). Conversely, excessive viscosity causes poor fuel atomization, which hinders combustion and lowers baked power.

The data acquired indicates that both catalyst compositions' flash points fall within the European Standard EN15940:2016/A1:2018 for green diesel. Because of its selectivity, which results in shorter chains, the 60Ni/γ-Al_2O_3 catalyst has lower flash point values. A lower flash point is implied by a shorter carbon chain, and the catalyst 15Ni-45Zn/γ-Al_2O_3 has the greater flash point findings. This is so because a product with a higher flash point is thought to have longer chains or fewer light fractions. With a hydrotreating procedure temperature of 400°C, the green diesel sample with the highest percent yield was used in this study. Based on Table 4, the cetane value generated in this study of 88.10 ± 0.648 CN and 82.33 ± 0.386 CN was achieved for 60Ni/γ-Al_2O_3, and 15Ni-45Zn/γ-Al_2O_3, respectively. With a minimum Cetane Number value of 70, this value complies with the Green Diesel European Standards EN15940:2016/A1:201. The selectivity of the catalyst itself has an impact on how the Zn promoter affects the cetane number results and the point at which the hydrotreating process lengthens the carbon chain. It is harder to burn carbon chains that are greater in length. Therefore, it influences the cetane number of the green diesel products produced. The 60Ni/γ-Al_2O_3 catalyst has a higher value than the 15Ni-45Zn/γ-Al_2O_3 catalyst, as can be observed from the data in Table 4 acid number. This is a result of a Zn promoter being added to lower the product's acidity level and functionalize the Zn promoter on the catalyst, resulting in a reduction in acidity. According to Gousi et al. (2020) has reported that the Zn promoter satisfied the minimum acid surface, while adding Zn can lower the acid number.

4. CONCLUSION

This study compares the 60Ni/γ-Al_2O_3 catalyst, both with and without a Zn promoter, to earlier studies that used the Ni-Mo/γ-Al_2O_3 catalyst. The 60Ni/γ-Al_2O_3 and 15Ni-45Zn/γ-Al_2O_3 catalysts are superior to the Ni-MO/γ-Al_2O_3 catalysts by green diesel criteria. The variations in catalyst materials have an impact on the final product's quality. Wherein the results of product selectivity and requirements vary depending on the component and mix. In addition to accelerating the reaction rate, the Ni-Zn/γ-Al_2O_3 catalyst has an impact on the final product's quality.

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