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Research Paper

Biodiesel Production from Palm Oil with Heterogeneous Catalysts

Jonathan[1], Samuel Gidan Rumpaisum[1,*], Rif'an Fathoni[1]

[1]Chemical Engineering, Universitas Mulawarman, Jl. Sambaliung No. 09 Kelurahan Gn. Kelua Samarinda, Postal Code 75119, East Kalimantan Indonesia

* Corresponding author: gidanrum@student.unmul.ac.id (Samuel Gidan Rumpaisum)

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Abstract: Rice production in East Kalimantan Province during 2023 reached around 215,290 tons/year of milled dry grain. Rice is one of the main agricultural products that produce waste in the form of rice husks. Rice husk is stillNot well utilized so that it only becomes a pile of agricultural waste. Rice husk ash contains compounds that can be used as catalyst support, including SiO2. This study aims to determine the effect of rice husk ash catalyst from the KOH impregnation method as a catalyst for making biodiesel from conventional palm oil. Transesterification was conducted on palm oil with the ratio of catalyst mass to oil volume (10%, 15%, 20%). Biodiesel production from palm oil using KOH impregnated catalyst with 2N concentration and 20% catalyst concentration produced the highest yield of 86.3%. Viscosity and density of the results obtained meet the standards.

Keywords: Biodiesel; Heterogeneous; Impregnation; Catalyst; Transesterification.

1. Introduction

Rice is the most important crop commodity in Indonesia, especially in Asia, according to data from the East Kalimantan Central Bureau of Statistics, rice production in East Kalimantan Province during 2023 reached around 215,290 tons of milled dry grain (MDG) [1]. Rice is one of the main agricultural products, besides being able to meet food needs, rice production also produces waste in the form of rice husks. Rice husk is one of the most beneficial agricultural wastes. However, farmers often assume that the husk can only be used as fertilizer, scouring ash, or animal feed [2]. The chemical content of rice husk ash, which has a SiO2 content of up to 80%, as well as other chemical content, can be used as a catalyst support in the biodiesel manufacturing process.

The need for energy, mainly fuel oil, continues to increase in line with population growth and technological advances. National fuel consumption continues to increase from year to year. Every day, national fuel consumption reaches an average of 140,000-180,000 kL. Devita (2015) explained that the increasing need for energy causes exploitation and excessive energy consumption, the higher the fuel consumption, the more depleted the petroleum reserves [3].

Biodiesel is one of the alternative fuels that can replace fuel oil and is produced by a chemical reaction between vegetable oil or animal fat with a short-chain alcohol, such as methanol, ethanol, or butanol, with the help of a catalyst, this process is called transesterification. From an environmental point of view, the use of biodiesel has several advantages, for example, it can reduce CO and CO_2

emissions, and is nontoxic and biodegradable. It is expected that biodiesel can reduce the use of fossil fuels [4].

Transesterification is the reaction of fat or oil with alcohol to form esters and glycerol. Catalysts are used to increase the reaction rate and yield. Commonly used alcohols are methanol and ethanol, Basic catalysts such as NaOH and KOH are more commonly used and accelerate the reaction significantly compared to acid catalysts, However, if the triglyceride contains more water and free fatty acids, transesterification with acid catalysts can be used [5].

Esterification reaction is a type of reaction that can be applied to produce biodiesel from fatty acid feedstock and methanol. In practice, the esterification reaction is slow and needs to be catalyzed by an acid catalyst. Acid catalysts are chosen to produce methyl esters with high free fatty acid content through esterification reactions. Acid catalysts such as H_2SO4, HPO_4, and HCl are effective catalysts for esterification reactions [6].

In previous studies, experiments have been carried out on the benefits of rice husks as catalysts in the process of making biodiesel such as research conducted by Kusyanto and Purwa Aditya in 2017, the raw materials used were rice husks and palm oil, the study varied the mass of rice husk ash catalyst, with the achievement of palm oil conversion into biodiesel by 67% [7].

In addition, research conducted by Pandiangan and Simanjuntak (2013) used raw materials of rice husk and palm oil. Rice husk was extracted (SiO_2) by sol gel method and then modified with MgO and used as heterogeneous catalyst in biodiesel production. The study varied the composition of the MgO/SiO2 catalyst and obtained the best results when the best conditions for the coconut oil transesterification process by the MgO/SiO2 catalyst were using a catalyst with a MgO/SiO_2 ratio of 1:5, at a temperature of 60^oC for 30 minutes. With the achievement of coconut oil conversion into biodiesel around 80% [8].

Impregnation is a stage that can improve the performance of rice husk ash catalysts. Impregnation involves the absorption or filling of rice husk ash pores with certain chemical substances. This can increase the surface area and absorption capacity of the catalyst, thus increasing its catalytic activity. Impregnation can be used to modify catalysts by adding or depositing certain compounds or metals into the pore structure or surface of calcined rice husk ash [9].

Therefore, the focus of the research is to utilize rice husk ash that has gone through calcination and impregnation processes as a heterogeneous catalyst in the production of biodiesel from palm oil.

2. Research and Methodology

2.1 Materials

This study was conducted to determine the effect of rice husk ash catalyst on the transesterification process in biodiesel production. In this study, rice husk that had been heated to ash was soaked with KOH solution through impregnation with different catalysts, namely 1N, 1.5N, and 2N. The transesterification process was carried out using a ratio of palm oil and methanol of 1:10 [10]. The concentration of rice husk ash catalyst used was 10%, 15%, and 20% of the mass of palm oil.

2.2 Catalyst Preparation Stage

The tools used in this research include furnace, erlenmeyer vacuum flask, magnetic stirrer, oven, Gegep, cup, spatula, analytical balance, condenser, beaker, hot plate, lumping pestle, 250 ml separating funnel, watch glass, and bulb. While the materials used include rice husk, Cruid Palm Oil (CPO), methanol, distilled water, KOH with concentrations of 1N, 1.5N, and 2N, rice husk ash calcined at 500°C for 3 hours, and rice husk ash calcined at 500°C for 3 hours with KOH impregnation concentrations of 1N, 1.5N, and 2N.

The rice husk was cleaned with water, then dried in an oven at 110°C until the weight was constant. A total of 100 grams of rice husk was put into an ash cup and heated in a furnace at 500°C for 3 hours. Afterwards, the rice husk ash was removed from the furnace and cooled in a desiccator for 15 minutes. The rice husk ash catalyst was soaked with 1N KOH for 24 hours, then heated until all the KOH was evaporated. The step was repeated for 1.5N KOH and 2N KOH concentrations.

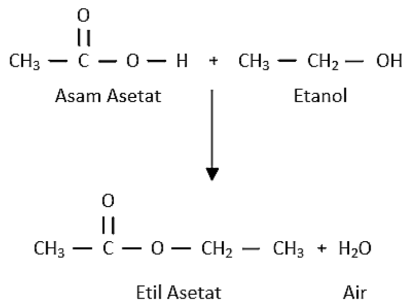
2.3 Determination of Free Fatty Acid Content

Weighing the sample as much as 5 grams into 125 ml erlenmeyer then adding 50 ml of ethanol that has been neutralized with 0.1NaOH. Added 5 drops of PP indicator to the sample. Titrate with 0.1NaOH which has been standardized until pink [11].

$$\%FFA = \frac{VNaOH \times NNaOH \times BM\ Free\ Fatty\ Acids}{Sample\ Mass \times 1000} \times 100\% \quad \ldots\ldots\ldots\ldots\ldots (1)$$

2.4 Esterification Stage

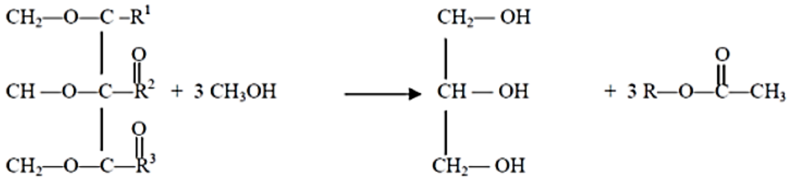
The CPO sample was heated and then weighed as much as 500 mL. Next, H2SO4 with a concentration of 1.5% was measured, and methanol was measured according to the ratio of 1:8. H2SO4 was mixed with methanol, then the solution was put into the CPO sample which had been heated to a temperature of 60°C. The heating process continued with a constant temperature of 60°C - 70°C for 4 hours. After that, the solution was allowed to stand until a layer formed for 24 hours. The top layer is then separated to be used in the next process, the transesterification reaction. Figure 1 is the mechanism of esterification reaction [12].



Figur 1. Esterification Reaction

2.5 Transesterification Stage

First, the top layer of esterification was taken and weighed as much as 100 ml. Then, 1N KOH/SiO2 catalyst was prepared with a catalyst concentration of 10% of the weight of the esterified top layer. Methanol was measured according to the ratio of 1:10, then put into the beaker along with the catalyst. Next, the solution was stirred for 2 hours using a magnetic stirrer. Palm oil that has been weighed as much as 100 ml is put into an Erlenmeyer/chemical beaker, and heated to a temperature of 64°C with a magnetic stirrer. After that, the methanol solution and catalyst were added to the heated palm oil, and the heating process was continued for 4 hours. Then, the solution was put into a separatory funnel and allowed to stand for 24 hours so that two deposits of biodiesel and glycerol were formed. The top layer suspected to be biodiesel was removed, and heated to 80°C to remove the methanol contained in the transesterification solution. This process was repeated using 1.5N, 2N KOH normality, and different catalyst concentrations (10%, 15%, 20%). The biodiesel yield was then calculated to obtain density, viscosity, and yield. Schematically, the transesterfication reaction with base catalyst can be seen in Figure 2 [13].



Figur 2. Transesterfication Reaction

The transesterification reaction of vegetable oil in the manufacture of biodiesel is outlined as follows:

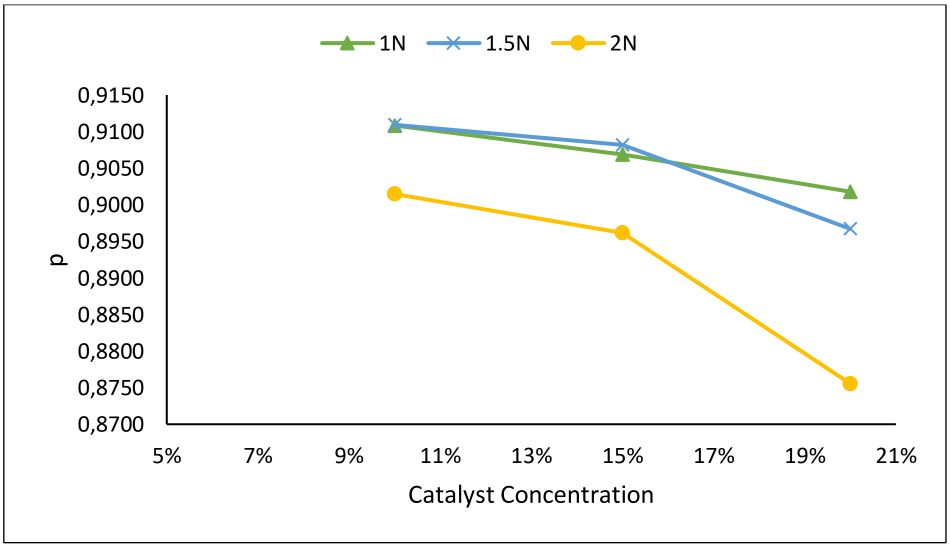
1. Vegetable oil is reacted with methanol through transesterification reaction to produce glycerin, methyl stearate and methyl oleate,
2. Methyl oleate (biodiesel) and glycerin are separated through a settling tank. After the glycerin is separated, the solution is washed with water, and then distilled to produce biodiesel according to the desired standard, the glycerin can be further processed for related industrial purposes.

3. Results and Discussion

3.1 Biodiesel Preparation

Before being used in the transesterification process, palm oil is first analyzed for free fatty acids. Free fatty acid content is an important factor because it can affect chemical reactions. High free fatty acids in raw materials can cause soap formation when alkaline chemicals are used as catalysts because alkaline chemicals react to neutralize free fatty acids from oil [14]. From the results of free fatty acid analysis, it was found that the free fatty acid content in the oil was 12.39% so that an esterification reaction was needed. The main purpose of esterification is to reduce free fatty acid levels in oil. Free fatty acid content in oil must be less than 5% to facilitate transesterification reaction. The test result of free fatty acid content in oil after esterification is 1.2% and has fulfilled to do transesterification reaction.

3.2 Effect of Husk Ash Catalyst Impregnation on Biodiesel Density



Figur 3 . Graph of the effect of husk ash catalyst impregnation on biodiesel density with concentrations of 10%, 15%, and 20%.

Figure 3 shows the graph at catalyst concentration of 10%; 15%; 20% with a temperature of 40°C. The density values at 1N catalyst impregnation with catalyst concentrations of 10%; 15%; 20% are 0.9108; 0.9068; 0.9018 g/mL, while at 1.5N catalyst impregnation the density is 0.9109; 0.9082; 0.8967 g/mL, while at 2N catalyst impregnation the density is 0.9015; 0.8962; 0.8756 g/mL. The largest density from the graph above is 0.9109 g/mL and the smallest density is 0.8756 g/mL. So that the density value that is in accordance with SNI standards is at 2N catalyst impregnation with 20% catalyst concentration of 0.8756 g/mL.

In Figure 4, it can be seen that the density value decreases inversely with increasing catalyst concentration, in accordance with the findings of Purwa and Hasmara (2017) stating that the more catalyst used, the lower the density value of biodiesel [7]. However, some points showed an increase in density value, which was caused by a decrease in temperature below the optimum operating temperature of 60°C-65°C during the transesterification process. This resulted in an increase in the density of the substance, as described in research by Wahyudi and Prayetno (2018), stating that temperature affects density, with lower temperatures resulting in higher density [15]. Permata Lestari (2021) confirms that the optimum reaction temperature is between 60°C-65°C [16].

In impregnation with 1N catalyst, the density value is stable decreasing because the temperature is maintained in the range of 60°C to 65°C, in accordance with the findings of Wahyudi and Prayetno (2018) The decrease in density also occurs at a concentration of 1.5N because the lower the density, the fewer components contained in it [15]. according to Prihanto and Irawan (2017), this can shorten the process of atomization of components when combustion occurs. The graph shows that 2N catalyst impregnation with 20% concentration produces the lowest biodiesel density, which is 0.8756 g/mL [17]. This shows that impregnation with 2N catalyst and 20% concentration is the optimum condition for transesterification in this study.

3.3 Effect of Husk Ash Catalyst Impregnation on Biodiesel Viscosity

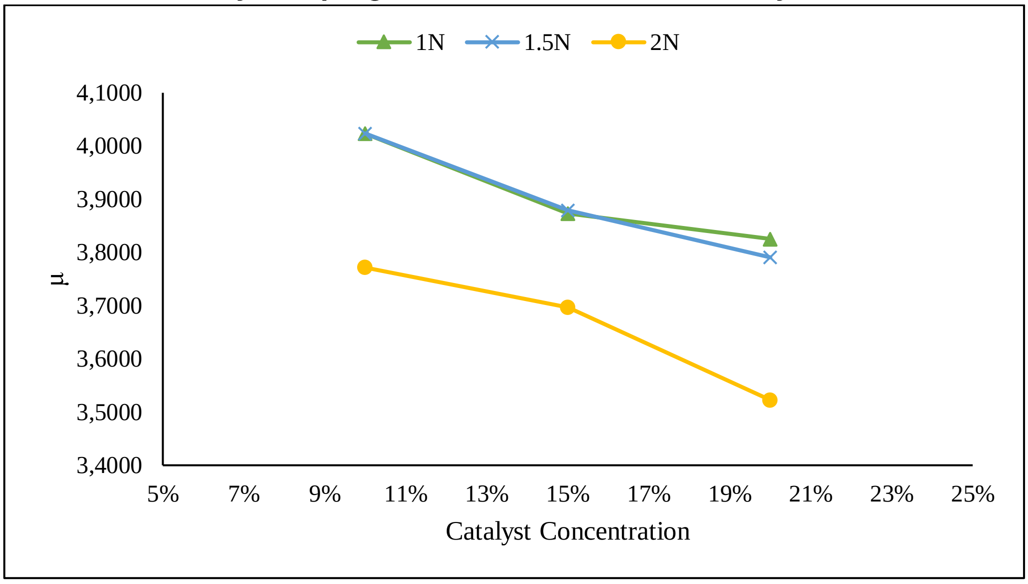


Figure 4. Graph of the effect of husk ash catalyst impregnation on the viscosity of biodiesel with 10%, 15%, and 20% concentrations

Figure 4 shows that at catalyst concentrations of 10%, 15%, and 20% at 40°C, the viscosity of biodiesel decreases as the catalyst concentration increases, which is consistent with the inverse trend. The viscosity values at 1N catalyst impregnation with 10%, 15%, and 20% catalyst concentrations were 4.0231 cSt, 3.8733 cSt, and 3.8254 cSt, respectively, while at 1.5N catalyst impregnation with the same catalyst concentration were 4.0235 cSt, 3.8791 cSt, and 3.7908 cSt, respectively. At 2N catalyst impregnation, the viscosities were 3.7715 cSt, 3.6970 cSt, and 3.5227 cSt, respectively. The range of biodiesel viscosity values is in accordance with SNI standards, which is 2.3-6 cSt, with the best viscosity occurring at the lowest value. An increase in viscosity can increase energy requirements for pumping and fuel injection, in accordance with the findings of [18]. The link between viscosity and density is that the greater the density between molecules, the higher the viscosity of the oil, according to [19]. The difference in viscosity values is due to incomplete separation, so some impurities, such as remnants of unreacted reactants, are still contained in the biodiesel, as explained by [18].

3.1 Effect of Husk Ash Catalyst Impregnation on Biodiesel Yield

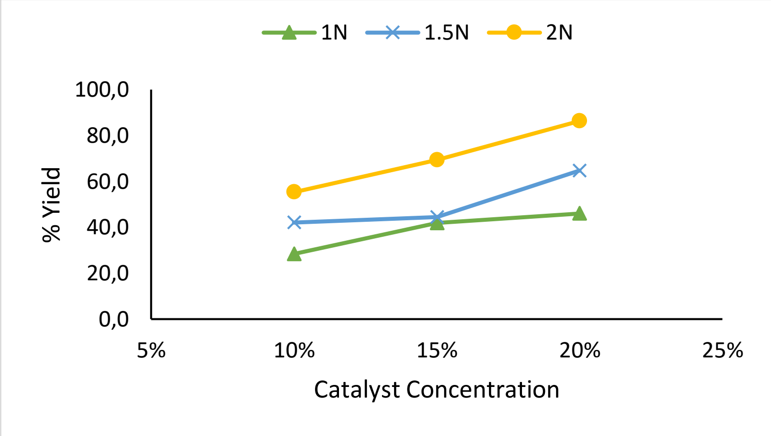


Figure 5. Graph of the effect of husk ash catalyst impregnation on Biodiesel yield with 10%, 15%, and 20% concentration.

Figure 5 shows that at 1N catalyst impregnation, the yield values with 10%, 15%, and 20% catalyst concentrations are 28.4%, 41.8%, and 46% respectively. At 1.5N catalyst impregnation, the yield values with the same catalyst concentration were 42.1%, 44.4%, and 64.7%. While at 2N catalyst impregnation, the yield values were 55.3%, 69.3%, and 86.3%. The highest yield, 86.3%, was achieved with 20% catalyst concentration at 2N catalyst impregnation, while the lowest yield, 28.4%, occurred with 10% catalyst concentration at 1N catalyst impregnation.

Figure 5 explains that the higher the catalyst concentration used, the greater the percentage yield of biodiesel, in accordance with previous research showing that increasing the catalyst concentration can increase the active side of the catalyst, and therefore increase the yield [7]. This confirms that the catalyst has a significant influence on the yield in the transesterification reaction of palm oil into biodiesel. The optimum condition in this study was reached at the variation of 2N catalyst impregnation with 20% concentration, which resulted in biodiesel yield of 86.3%, density of 0.8756 g/mL, and viscosity of 3.5227 cSt.

4. Conclusion

Based on the results of the data discussed, it can be concluded that the manufacture of biodiesel using heterogeneous catalysts is carried out through the esterification process followed by transesterification. The density value decreases as the catalyst mass increases, with the largest density of 0.9109 g/mL and the smallest density of 0.8756 g/mL. Density that is in accordance with SNI standards is at 2N catalyst impregnation with 20% catalyst mass, which is 0.8756 g/mL. Viscosity decreased with increasing catalyst mass, where the best viscosity according to SNI standards was at 2N catalyst impregnation with 20% catalyst mass, which was 3.5227 cSt. According to experts, too high viscosity can affect the performance of fuel injection equipment and make it difficult to fog fuel oil. The highest yield value was achieved by using 20% catalyst mass at 2N catalyst impregnation of 86.3%, while the lowest yield was produced by using 10% catalyst at 1N catalyst impregnation of 28.4%. The higher the mass of catalyst used, the greater the percent yield of biodiesel products produced.

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