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Utilization of Low-Grade Ceiba Pentandra Oil for Biodiesel Production Using Nano Zinc Oxide Catalyst Supported by Fly-Ash Waste

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ARTICLE INFORMATION

Journal of Science and Technology – Volume 28 Number 1, May 2024

Page:
75 – 84
Date of issue :
May 31, 2024

DOI:
10.31284/j.iptek.2024.v28i1.5937

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PUBLISHER

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ABSTRACT

Biodiesel is a renewable energy and potentially to be developed to replace fuel derived from fossil oil. This study explores the oil of Ceiba pentandra (OCP), a low-quality oil with high free fatty acids as a feedstock for biodiesel production. A cost-effective nano-doped zinc oxide heterogeneous catalyst was developed using fly ash waste as a support called NZO/FA. The catalyst was synthesized using co-precipitation, precipitation, and impregnation methods. Biodiesel is made through a transesterification process using a laboratory-scale glass-type batch reactor. The experiment examines how the varied temperature (60-80°C with an interval of 10°C) and varied reaction time (3-5 hours with an interval of 0.5 h) influence biodiesel yield, heating value, and final product properties. Meanwhile, other conditions were fixed at a molar ratio of oil/methanol of 1:15 and a catalyst dosage of 5%. The results showed the highest yield of 98.69%, and a GHV of 37.95 MJ/kg was obtained at a reaction temperature of 80°C and a reaction time of 5 hours. Meanwhile, almost all of the characteristics of the biodiesel produced meet SNI 7182:2015.

Keywords: Ceiba pentandra oil; biodiesel; fly ash; gross heating value; heterogeneous catalyst.

ABSTRACT

Biodiesel merupakan salah satu energi terbarukan yang sangat berpotensi untuk dikembangkan untuk menggantikan bahan bakar yang bersumber dari minyak fosil. Tujuan penelitian ini adalah untuk memanfaatkan minyak biji kapuk (OCP) yang termasuk minyak berkualitas rendah (LGO) dengan kandungan asam lemak bebas (FFA) tinggi (10%) sebagai bahan baku pembuatan biodiesel menggunakan katalis hetrogen nano seng oksida dengan penyangga murah dari limbah abu terbang (NZO/FA). Katalis disintesis menggunakan metode ko-presitasi, presipitasi dan impregnasi. Pembuatan biodiesel melalui proses transesterifikasi menggunakan reaktor batch tipe glass berskala laboartorium. Faktor-faktor yang dipelajari meliputi pengaruh temperatur dan waktu transesterifikasi terhadap yield biodiel, gross heating value (GHV) dan karakteristik biodiesel yang dihasilkan. Proses transesterifikasi dilakukan pada temperatur yang bervariasi mulai 60-80°C dengan interval 10°C, waktu reaksi bervariasi 3-5 jam dengan interval 0,5 jam. Sedangkan kondisi lainnya konstant pada rasio molar minyak /metanol 1:15 dan dosis katalis 5%. Hasil penelitisan menunjukkan yield tertinggi sebesar 98.69%, dan GHV sebesar 37,95 MJ/kg diperolah pada temperaturi 80°C dan waktu reaksi 5 jam. Sedangkan karakteristik biodiesel yang dihasilkan hampir semuanya memennuhi SNI 7182:2015.

Keywords: *abu terbang; biodiesel; minyak biji kapuk; katalis heterogen; nilai kalor.*

INTRODUCTION

Our global reliance on fossil fuels, a finite resource, creates a looming problem. As energy demands continue to rise yearly, we face the challenge of finding sustainable alternatives [1] [2]. Data from the Ministry of Energy and Mineral Resources [3] reveals a significant rise in fuel consumption to 75.27 million kiloliters in 2021. This highlights the urgent need to address energy sustainability challenges to prevent future energy crises [3]. However, fossil fuels come at a significant environmental cost. Their burning contributes to global warming, amplifies the greenhouse gas effect, and pollutes the air [1] [2]. Investing in renewable energy technologies offers a promising solution to these interconnected challenges [1].

Biodiesel emerges as a promising alternative to conventional fossil fuels. Its environmental benefits include reduced combustion emissions compared to traditional fuel sources [4] [5]. *Ceiba pentandra* oil (OCP) emerges as a compelling feedstock for biodiesel production. Derived from vegetable oils through a chemical process (transesterification), biodiesel offers a sustainable alternative to fossil fuels [1]. Indonesia's extensive C*eiba pentandra* (CP) plantations (7,630 ha) make OCP a readily available resource. Additionally, CP seeds boast a high oil content (25-40%) rich in unsaturated fatty acids (63.27%) ideal properties for biodiesel production. Crucially, kapok is classified as a non-edible oil, ensuring that OCP production wouldn't compete with food crops, and addressing food security concerns [6] [7].

Non-edible oils often contain high levels of free fatty acids (FFA), posing a challenge for biodiesel production. To address this, researchers have turned to solid/heterogeneous catalysts. These catalysts offer a more efficient and cost-effective solution. They can handle FFA while simultaneously carrying out both esterification and transesterification reactions, reducing processing steps. Additionally, they eliminate the need for product washing, further streamlining the biodiesel production process [8]. Heterogeneous catalysts offer significant advantages in biodiesel production. They are easily separated from the final product, allowing for reuse in subsequent cycles. Additionally, they prevent soap formation as a byproduct, streamlining the purification process [1] [2] [3] [8]. This study explores a nano zinc oxide catalyst supported by fly ash (NZO/FA) for biodiesel production from OCP.

This study utilizes Zinc Oxide (ZnO) as the catalyst due to its exceptional qualities. ZnO acts as a powerful heterogeneous acid catalyst across its entire surface area. It's also non-toxic, reusable, easily separated, and environmentally friendly, making it an attractive choice. Notably, ZnO achieves an impressive biodiesel yield of 86.1% [5]. Fly Ash (FA) is incorporated as a support material to simplify catalyst separation. Its high silica content increases the reaction surface area, accelerating biodiesel production. Furthermore, FA is eco-friendly and readily available in Indonesia as a byproduct of coal combustion in power plants [9]. [10] synthesized and characterized NZO/FA catalysts without chemical treatment on the fly ash. This resulted in a less active FA surface, hindering the catalyst's performance. The highest biodiesel yield achieved during OCP transesterification was just 51.01%. This limitation might be attributed to the uneven distribution of ZnO, which functions as a promoter within the catalyst, across the FA surface. Asri et al. (2023) [11] further continued the development of the NZO/FA by modifying the route of catalyst synthesis by subjecting treatment to the FA before synthesizing to be catalyst support. After characterizing using SEM, XRF, XRD, BET, and FTIR methods the catalyst showed a significant improvement in the characteristic performance in which ZnO particles were well dispersed in the surface of FA. Activity tests were carried out with the same oil (OCP) and under the same conditions as previous research [10] showed that biodiesel yield was very significantly increased by 95.8% [11]. Therefore, in this work, we use the same route and conditions to synthesize the NZO/FA catalyst to convert OCP into biodiesel. The study focuses on optimizing the transesterification factors including temperature, and reaction time on biodiesel yield. Moreover, we also investigate the influence of those factors on gross heating value (GHV) and the characteristics of biodiesel produced.

LITERATURE REVIEW

Heterogeneous catalyst

A heterogeneous catalyst is a catalyst that is in a different phase from the reactants. It has some advantages such as being reused, the ease of separation of FAME and glycerol, and the low waste produced. Heterogeneous catalysts are widely used in various fields including transesterification processes such as alkaline earth metal catalysts, hydrotalcite, mixed metal oxides, and resin ions. Catalytic activity can be increased using promoters or supports [12].

Fly ash

Fly ash is waste from coal-fired steam power plants (PLTU). The State Electricity Company (PLN), on average produces 5,234,400 tons/year of fly ash and bottom ash annually, of which 20% (1,046,880 tons) is fly ash [11]. This waste includes dangerous so its accumulation can damage the environment. Meanwhile, fly ash is only used as concrete or artificial aggregate to produce asphalt. Developing efficient environmentally friendly applications is very important, for example, producing cheap friendly heterogeneous catalysts to produce biodiesel. The possibility due to its chemical composition (dray base) is SiO2 (50.91%), Al2O3 (30.91%), Na2O 0.10%, CaO 6.2%, Fe_2O_3 3.46%, MgO 1.48%, TiO_2 1.65%, MnO 0.02%, K_2O 0.60%, P_2O_5 0.56% (Babajide, 2010)

Transesterification Reaction

Transesterification is a reaction between triglycerides and short-chain alcohols that produces glycerol as a byproduct. Meanwhile, the three triglyceride fatty acid chains will be freed from the glycerol framework, and when interacting with short-chain alcohols, they turn into biodiesel [13].

Biodiesel

Biodiesel is a fuel made from plant oils and animal fats. It's a promising alternative to regular diesel because of its environmentally friendly nature. Biodiesel is non-toxic, decomposes naturally, and doesn't contain harmful substances like aromatics. It also reduces emissions of sulfur oxides and soot particles from engines [14].

Gross Heating Value (GHV)

An important factor in choosing a fuel is its energy content, measured as heating value. This heating value is determined by burning a controlled amount of fuel in a special chamber and capturing all the heat released. This method, using an oxygen bomb calorimeter, accounts for even the energy stored in the water vapor produced during combustion. Because it reflects the total energies available from the fuel, heating value is one of the crucial properties for evaluating its usefulness. Gross Heating Value (GHV) is the amount of energy released by fuel in the complete combustion process per unit of mass under standard conditions [15].

METHOD

The process involves four key stages: pre-treatment/activation of fly ash as support, synthesis of NZO/FA catalyst, pretreatment and characterization of OCP, and optimization of the transesterification process.

Fly Ash Activation:

Fly ash, intended as a support material, undergoes pre-treatment with hydrochloric acid to increase its porosity. This activation step enhances the adhesion of zinc oxide (ZnO), the active component, onto the fly ash surface. Asri et. (2023) [11] describe the specific conditions involved in this activation (0.5 M HCl, 70°C, 1-hour stirring). The pre-treatment effectively activates and opens the pores within the fly ash (FA) allowing for better adhesion of zinc oxide (ZnO) on the catalyst's surface. This enhanced adhesion contributes to the high yield achieved as described by [16].

NZO/FA Catalyst Synthesis

Based on prior research, the optimal parameters for synthesizing the NZO/FA catalyst are employed in this step. These parameters include a 50% ZnO loading and a calcination process at 500°C for 5 hours. These conditions were identified to yield a catalyst with the most desirable characteristics [11].

Pretreatment and Characterization of OCP.

It is well known that OCP is a low-grade oil with a high content of gum, FFA, phosphatides, and other impurities that can interfere with the transesterification process. The degumming process aims to achieve good quality OCP which is resistant to oxidative degradation, rancidity, and incomplete combustion processes when oil is used as fuel [17]. In addition, good-quality oil will increase the yield of biodiesel (Mirzayanti et al., 2020). Therefore, before the transesterification process, the oil must be treated through a degumming process with 0.1% phosphoric acid (wt.% of the oil) using the procedure as described in the previous research [11]. Characterization of OCP was done before and after treatment.

Transesterification Process Optimization

The final stage is converting OCP into biodiesel using NZO catalyst to optimize the transesterification process parameter. In this work, we investigate the influence of temperature and time of transesterification on biodiesel yield. Furthermore, the effect of those parameters was also observed on the GHV biodiesel. The experiment was conducted in a set of laboratory-scale glass-type batch reactor equipment. The optimization parameter process was done at varying temperatures from 60 to 80°C (with 10°C), and reaction time varies from 3 to 5 h with 0.5 h of intervals. Meanwhile, the other conditions were fixed at 5% of the catalyst dose, and 1:15 molar ratio of OCP to methanol. The complete transesterification procedure refers to previous studies [11] [12] [19]. The density and volume of the biodiesel produced were measured at each treatment. These data are used to calculate the mass of biodiesel produced. Further, the yield of biodiesel was calculated by Equation (1). The highest biodiesel yield at each variable was then analyzed for its GHV. Finally, researchers analyzed various properties of the biodiesel produced under ideal conditions, including density, viscosity, acid content (FFA), degree of unsaturation (iodine number), and combustibility (cetane number).

$$yield\ (\%) = \frac{W_{BD}}{W_0} \times 100\% \qquad \text{..... (1)}$$

where, W_{BD} is the weight of biodiesel (g) obtained in experiments, while Wo is the weight of kapok seed oil (g) used in experiments.

RESULTS AND DISCUSSION

OCP characteristics

The fatty acid composition of kapok seed oil was analyzed using GC-MS and it was found that the highest fatty acid content is linoleic acid (59.1%), palmitic acid (28.51%), and stearic acid (9.59%). It means that OCP was dominated by 59.1% unsaturated fatty acid. OCP was also characterized either before or after treatment. To determine the effectiveness of the treatment process (degumming process), the OCP was characterized both before and after treatment. The FFA content of OCP was calculated based on linoleic acid due to its dominance. The result of OCP characterization is shown in Table 1. It shows that the quality of OCP after degumming is better than before, with FFA content, density, saponification value, and moisture content decreasing to 9.6 %, 0.910 g/mL, 223, and 0.05%, respectively. Meanwhile, the Iodine number increases to 72.94 gr I_2/100 gr. Furthermore, the treated OCP visually becomes clear and clean with yellowish-gold color.

Table 1. Characteristics of OCP

Parameters Test	Value	
	Before Pre-Treatment	After Pre-Treatment
Linoleic Acid as FFA (%w/w)	12.84	9.6
Iodine Number (gr I_2/100 gr)	61.47	72.94

Parameters Test	**Value**	
	Before Pre-Treatment	**After Pre-Treatment**
Saponification Value	279	223
Density (gr/mL)	0.95Value	0.91
Viscosity (cSt)	2.19	2.23
Moisture Content (%w/w)	0.14	0.05
Color	Dark yellowish	Clear golden yellow

Optimization of transesterification parameters

Many factors influenced the transesterification process including temperature, time reaction, molar ratio oil to methanol, catalyst dose, and mixing speed. In this work, we focus on optimizing temperature and reaction time. The experiment was conducted at three different temperatures (60. 70 and 80°C) and varying reaction times of 3; 3.5; 4; 4.5 and 5h. The effect of those variables on the yield of biodiesel is shown in Figure 1. The study found a positive correlation between reaction time and biodiesel yield at all temperatures tested. At 3h of reaction time, biodiesel yield at 60, 70, and 80°C is 65.45, 78.15 and 87.75%, respectively. Biodiesel yields are relatively low compared with the longer time use. It is possibly due to the lack of time needed to convert more oil into biodiesel. However, the effect of reaction temperature on biodiesel yield is greater than the effect of the reaction time. Theoretically, according to Arrhenius's law, the temperature exponentially affects the reaction rate constant, which is the enhancing rate of reaction. The reaction temperature significantly impacts the reactant molecules and ultimately influences the reaction rate because the temperature is directly proportional to the average kinetic energy of the molecules. The heat up the reaction mixture, the solubility of methanol increased, enhancing the reactant molecules to move faster, and then collide with each other more frequently [20] [21]. In this work, the highest yield of 98.69% was obtained at 80°C at 5 h reaction time.

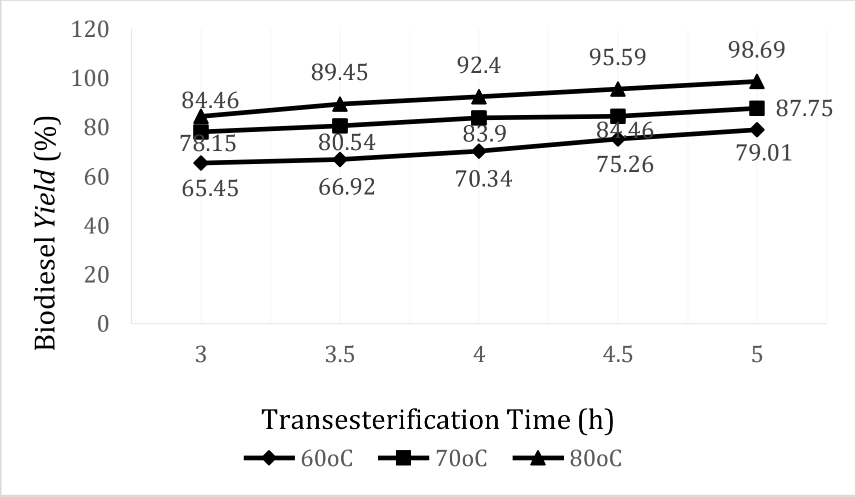

Figure 1. Influenced time reaction on biodiesel yield at various temperature reaction

This study achieved a significantly higher biodiesel yield compared with previous research [22] [23]. Yusuff et al. [22] reported a maximum yield of 83.17%, despite using a higher reaction temperature of 140°C. Similarly, Babajide et al. [23] only achieved a maximum yield of 86.13%, even at a much higher temperature of 160°C. It was proven that the pretreatment carried out on FA before it was synthesized into NZO/FA had a significant effect on catalyst activity

Gross Heating Value (GHV)

GHV relates to the amount of energy that can be produced from the burning process of fuel. **GHV** depends on the fuel's chemical makeup, particularly the amount of carbon and hydrogen contained (since these contribute most to the energy released) [24]. The burning process itself is a chemical reaction between the fuel and oxygen. The gross heating value sometimes called the higher heating value, considers all the heat released, including the energy used to vaporize any water created during combustion. This assumes the water vapor condenses and releases its latent heat outside the system. The net heating value, also known as the lower heating value, represents the usable heat you can extract after accounting for the energy carried away by water vapor in the exhaust.

In this study, GHV was tested at the optimum conditions at each temperature involved (at 5 h reaction time), and the results are shown in Table 2.

Table 2. GHV analysis results

Transesterification temperatures (°C)	GHV (MJ/kg)
60	37.61
70	37.68
80	37.95

There are two main types of gross heating value (GHV): higher heating value (HHV) and lower heating value (LHV). Biodiesel is considered HHV if its GHV falls between 39-43.33 MJ/kg. Biodiesel with a heating value below 39 MJ/kg falls under the LHV category (Dirgantara et al.2019). It is shown that the GHV value falls in the range of 37.61 and 37.95 MJ/kg, which means that the caloric value of OCP's biodiesel follows the LHV categories. According to ASTM D15 standards, biodiesel typically has a heating value between 35-40 MJ/kg. This indicates good quality fuel with several advantages: high heating value, longer burn time, low ash and moisture, and complete combustion. Conversely, a low heating value might indicate the presence of water vapor (H_2O) in the biodiesel [25][26].

OCP's Biodiesel Characteristics

The characteristics of biodiesel must be rigorously tested, to guarantee that the biodiesel functions well with engines and doesn't harm them, This testing safeguards not only the engine's health but also environmental benefits, compliance with regulations, and efficient production. In this study, we followed the standard SNI 7182:2015 methods to assess crucial physical and chemical properties of the biodiesel, such as free fatty acid content (FFA), iodine number, saponification value, density, moisture content, and cetane number. The characteristics of biodiesel are tabulated in Table 3. It is shown that the overall characteristics meet the standard SNI 7182:2015, except the FFA content is slightly higher than the standard, due to the initial content of FFA in OCP being too high (mostly 10%) [27]. An iodine number of 74.08 g I2/100g for biodiesel (it still falls within the standard) indicates a relatively high degree of unsaturation in the fatty acid chains that compound the biodiesel, it matches the raw material used, namely OCP which is dominated by unsaturated fatty acids. In some cases, a slightly higher iodine number might contribute to better cold start performance due to the lower cloud point. In addition, unsaturated compounds can increase performance due to an increasing the cetane number [28][29]. Saponification value (SV) is the amount of potassium hydroxide (KOH) needed to completely saponify (convert to soap) one gram of fat or oil. A higher SV indicates a lower average molecular weight of the fatty acids and potentially a higher proportion of short-chain fatty acids. It provides clues about the potential fatty acid profile of the feedstock used for production.

Density is another important property of biodiesel. It means the weight of a unit volume of fluid. Since engine fuel injection relies on a volume-based measurement system, denser biodiesel will deliver a slightly higher fuel mass for the same volume. However, this isn't always positive. Excessive density can cause wear and tear on the engine. On the other hand, **a** very low density might indicate the presence of leftover incomplete separation methanol or other impurities in the biodiesel. This is suggested during the production process [30]. Viscosity, a measure of a fuel's resistance to flow, significantly impacts the engine's performance. It regulates mass transfer and metering essential for proper engine operation. High-viscosity fuels have lower volatility and poor atomization during injection in compression ignition (CI) engines. This can lead to incomplete combustion, and ultimately cause carbon deposit**s** on the injector nozzle and within the combustion chamber.

Meanwhile, the high moisture content significantly compromises the quality and performance of biodiesel. The high moisture content could significantly compromise the quality and performance of biodiesel. For example, it reduces heating value meaning less energy is released during burning. In addition, moisture reacts with sulfur in the fuel, forming acids that corrode engine components. It also promotes a chemical reaction (hydrolysis) that reduces the final amount of usable biodiesel produced. Furthermore, moisture can cause the biodiesel to foam, creating air pockets that hinder efficient combustion. Foam and water provide a breeding ground for microbes, leading to impurities that can harm the engine [31]. When compared to previous research [10-11], the characteristics of biodiesel in this study are much better for all attributes (FFA content, Iodine content, Saponification value, density, viscosity, and moisture content). When compared with previous research, the characteristics of biodiesel are even better, especially for the density and viscosity attributes [22].

Table 3. Characteristics of OCP’s biodiesel at optimum conditions.

Parameters Test	SNI 7182:2015	Analysis Findings
Linoleic Acid as FFA (%w/w)	0.6, max	0.7
Iodine Number (gr I_2/100 gr)	115, max	74.08
Saponification Value	189-197	189
Density (gr/mL)	0.85-0.89	0.86
Viscosity (cSt)	2.3-6.0	2.33
Moisture Content (%w/w)	0.05, max	0.0002

CONCLUSION

This study successfully optimized the transesterification process of Ceiba pentandra oil using a cost-effective nano zinc oxide catalyst supported by fly ash waste (NZO/FA**).** Temperature and time reactions were identified as crucial factors significantly impacting biodiesel yield. Higher temperatures and longer reaction times generally led to increased yield, with temperature having a more pronounced effect. The optimal conditions achieved a remarkable biodiesel yield of 98.69% at 80°C for 5 hours, with a methanol-to-oil ratio of 1:15 and a 5% catalyst dose. Furthermore, the produced biodiesel exhibited a gross heating value (GHV) within the range of 37.61-37.98 MJ/kg, falling under the LHV category and complying with ASTM D15 standards**.** Additionally, the biodiesel characteristics met the requirements of SNI 7182: 2015. These results demonstrate the promising potential of the NZO/FA catalyst for efficiently converting Ceiba pentandra oil into high-quality biodiesel.

ACKNOWLEDGMENT

Thank you to the Directorate General of Higher Education, Research, and Technology of the Republic of Indonesia and Research and Community Services Institutions of Universitas Ciputra Surabaya for funding this research through a PDUPT grant with contract number

013/SP2H/PT/LL7/2022, and DIP-BIMA grant with contract No: 007/UC-LPPM/DIPBIMA/SP3H/IX/2023.

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