JURNAL IPTEK
MEDIA KOMUNIKASI TEKNOLOGI
homepage URL : ejurnal.itats.ac.id/index.php/iptek

Transesterification of Kesambi Seed Oil (*Schleicher oleosa L*) With Zinc Oxide Catalyst (ZnO) Supported with Multi-Wall Carbon Nanotubes (MWCNTs)

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ARTICLE INFORMATION

Jurnal IPTEK – Volume 26
No. 1, May 2022

Page:
41 – 49
Published Date :
31 May 2022

DOI:
10.31284/j.iptek.2022.v26i1.2967

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ABSTRACT

The alternative energy that has been developed today of which is biodiesel. Biodiesel is a fuel that processes from updated renewable sources. Kesambi seed oil is one source that has the potential to be used as a raw material. This research focuses on developing ZnO catalysts with MWCNTS support for the transesterification process of kesambi seed oil. This study investigates the type of sulfonation time, impregnation time, and length of stirring time during the transesterification process to biodiesel product results. Catalysts are characterised by X-Ray diffraction analysis (X-RD), Brunauer Emmett-Teller (BET), scanning electron microscopy energy dispersive X-Ray spectroscopy (SEM-EDX). The results showed the highest marks of 85.46% were obtained at a variable impregnation time of 12 hours, a sulfonation time of 4 hours with a calcination temperature of 500°C, a calcination time of 4 hours, a molar ratio of oil to methanol of 1:15, a transesterified reaction temperature of 65 ° C, and a stirring time length on a transification reaction of 7 hours.

***Keywords**: Biodiesel, Kesambi seed oil, Zinc oxide, MWCNTs, Transesterification*

ABSTRACT

Energi alternatif yang telah dikembangkan saat ini, salah satunya adalah biodiesel. Biodiesel adalah bahan bakar yang diproses dari sumber terbarukan yang diperbarui. Minyak biji Kesambi merupakan salah satu sumber yang berpotensi untuk digunakan sebagai bahan baku. Penelitian ini berfokus pada pengembangan katalis ZnO dengan dukungan MWCNTS untuk proses transesterifikasi minyak biji kesambi. Tujuan dari penelitian ini adalah untuk mengetahui jenis waktu sulfonasi, waktu impregnasi, dan lamanya waktu pengadukan selama proses transesterifikasi terhadap hasil produk biodiesel. Katalis dicirikan oleh analisis difraksi sinar-X (X-RD), Brunauer Emmett-Teller (BET), pemindaian spektroskopi sinar-X dispersif energi mikroskop elektron (SEM-EDX). Hasil penelitian menunjukkan hasil tertinggi sebesar 85,46% diperoleh pada variabel waktu impregnasi 12 jam, waktu sulfonasi 4 jam dengan suhu kalsinasi 500 c, waktu kalsinasi 4 jam, rasio molar minyak terhadap metanol 1:15, suhu reaksi transesterifikasi 65 C, dan lama waktu pengadukan pada reaksi transifikasi 7 jam.

Kata kunci: Biodiesel, Minyak Biji Kesambi, Seng oksida, MWCNTs, Transesterifikasi

INTRODUCTION

The population increase in the population on earth has resulted in the increasing number of fossil fuel consumption needs. It is accompanied by technological developments resulting in increasing energy needs that cannot be updated. So far, the fuel energy sources that are most often used in life are fossil fuels, while the use of fossil fuels is increasingly thinning and, if used continuously and excessively, will create energy scarcity in the future. Many alternative energy sources such as biodiesel, biogas, and biomass have been developed. One of the alternative energy sources that many developed researchers today is biodiesel. Biodiesel is a fuel processed by updated sources generally from oil in plants or animal fat [1]. One source of raw material for biodiesel production that has the potential to be developed is kesambi oil because the oil content is relatively high, around 70-73% of dry seeds [2]. The existence of kesambi oil is relatively abundant because the kesambi plant is being cultivated by the ministry of foresty. However, kesambi oil is non-edible and cannot be used in food processes. In addition, it has high levels of free fatty acids and is still far from the SNI standard. High levels of free fatty acids make oil tend to be easily damaged due to environmental influence. Damaged fats, if consumed, can cause health problems such as obesity, poisoning in the body, increased risk of cancer, risk of degenerative diseases, and deposition of fat cells in organs such as arteries, kidneys, liver, and heart [3]. Therefore, it is used as a raw material for biodiesel.

The process of making biodiesel can take two steps: esterification and transesterification. The esterification process aims to reduce the oil or fat used [4]. In contrast, the transesterification process is the transformation of large triglyceride molecules, branching from vegetable oil and grease into smaller, straight-chain molecules, and almost the same as inner molecules of Diesel fuel [5]. A catalyst is needed to accelerate the reaction rate in biodiesel so that the reaction conversion will be achieved quickly. Homogeneous catalysts are chosen because they produce higher methyl esters with a relatively short reaction time. On the other hand, the homogeneous catalyst has several disadvantages, such as the formation of a side product in the form of soap can reduce the biodiesel yield and make it difficult for biodiesel separation with catalysts. The corrosive and waste can pollute the environment [6]. Other disadvantages of homogeneous catalysts include catalyst recovery problems, large amounts of wastewater, and glycerol quality problems as a side product [7]. Developing heterogeneous catalysts into the right solution to overcome issues in homogeneous catalysts, heterogeneous catalysts have easily separated properties of products. They can be reused even though they repeatedly reduce biodiesel production costs. In addition, the waste of alkaline products such as soap will not be formed, and glycerol with high purity (more than 98% WT) produced has a phase that is different from the main product so that it is easily separated [8].

Currently, many heterogeneous catalysts have been developed, one of which is zinc oxide; the selection method of zinc oxide as the main active component is because the biodiesel produced with ZnO catalysts has a yield of 86.1% and the high methyl ester content is 98.9%. This shows that the ZnO catalyst makes biodiesel products with a very high level of purity [9]. But because of the small size of the zinc oxide, it causes difficulty in the process of separation of the catalyst with the product of transesterification, so support is needed so that the process of separation of the trigger with the trans transition product becomes easier to do. The selection of MWCNTS as a support will be thoroughly dispersed in the ZnO catalyst component. In addition, MWCNTS has several intrinsic properties that are beneficial for catalysis, e.g. high surface area, high purity, surface morphology, and well-developed porosity [8].

In previous research, transesterified biodiesel seed oil with ZnO catalysts and supported by multi-walled carbon nanotube (MWCNTS) by Asri et al. (2020) [10]. It was reported that the results obtained were unsatisfactory; ZnO spread in the pores, so the results of biodiesel products were not optimal. To increase catalyst activity, researchers modified synthetic catalyst routes by adding sulfonate groups and ammonium sulfate during sulfonation within a specified time. MWCNTS researchers hope to become more reactive so that ZnO can stick to the surface of MWCNTS and the resulting biodiesel can be maximised. Therefore, this study added a sulfonation process using ammonium sulfate to hope that the MWCNT surface is more reactive so that ZnO can stick well on the surface. Some of the parameters that will be studied include the effect of sulfonation time (h), impregnation time (h) and transesterification time (h) on biodiesel yield, respectively.

LITERATURE REVIEW

Biodiesel

Biodiesel is another type of fatty ester, generally in the form of a monoalkyl ester that uses vegetable or animal oils with physical processes and chemicals mixed with a suitable composition [11]. In addition, biodiesel is also called green energy because it is a renewable, biodegradable and environmentally friendly fuel oil that does not significantly increase global warming [12]. The advantages of biodiesel include having high cetane numbers, non-toxic, safe and high lubricating power.

Catalyst

Catalysts accelerate the rate of a chemical reaction at a specific temperature without altering or being used by the response itself. The trigger is divided into two: homogeny catalyst and heterogeneous catalyst; homogeny catalyst is used when the motivation has the same phase. At the same time, the heterogeneous catalyst is used if the stage is different from the reactant. In this study, the ZnO zinc oxide catalyst is heterogeneous because biodiesel produced using ZnO catalyst has high purity.

Transesterification Reaction

The transesterification reaction is the chemical transformation of large triglyceride molecules branched from vegetable oils or animal fats into smaller molecules, molecules with straight chains, and almost the same as in diesel fuel. Vegetable oils or animal fats react with alcohols (methanol) with the help of catalysts that produce alkyl esters (or, for methanol, methyl esters) [5]. Several factors can influence the transesterification reaction, such as a) type of raw material, b) type of alcohol, c) catalyst, d) mole ratio of oil: methanol, e) reaction temperature, f) reaction time, and g) stirring [6].

MATERIAL AND METHODS

Materials and Equipment

The tools used are Burette, beaker glass, porcelain cup, oven, furnace, measuring flask, measuring cup, separator funnel, Erlenmeyer, timer, hotplate stirrer, three-neck flask, three-neck flask stopper, water bath, thermometer, condenser, filter paper Whatman no. 40, volume pipette, pycnometer, weigh bottle & viscometer Ostwald.

The reagents used are analytical grade quality for Catalyst synthesis, namely zinc acetate, oxalic acid, sodium hydroxide, sodium iodide, potassium hydroxide, sodium thiosulfate, hydrochloric acid, acetone, and methanol using Merck products, Germany. SAP Chemical supplies other reagents such as ammonium sulfate and phosphoric acid. Meanwhile, MWCNTs carbon nanotube walls are provided by Advanced Materials Esoterica, China [13]. In addition, technical grade methanol as raw material for biodiesel synthesis is provided by nearby chemical providers.

Methods

This research is a laboratory-scale experimental study, namely the process of making zinc oxide and MWCNTS as a supporting medium for transesterification of kesambi oil. The implementation of research includes oil conditioning, catalyst preparation, catalyst characterisation, and transesterification process.

Kesambi oil conditioning is carried out with the preparation stage of the initial oil analysis before use (pre-treatment), then separating the sap’s mucus (degumming). After that, the oil will be analysed again, and then the process of decreasing free fatty acids (FFA). The final stage of conditioning the Kesambi oil is mechanising the Kesambi oil after a decrease in free fatty acids (FFA).

While preparing catalysts, the manufacture of ZnO/MWCNTS catalysts is carried out. ZnO/MWCNTS was synthesised by the stober-like mechanism modified by researchers in previous studies, followed by precipitation and additional steps starting from sulfonation steps as a new route [14]. First, the MWCNTS surface must be activated by the -SO3H group using ammonium sulfate. This sulfonation step aims to use the ZnO catalyst as a metal oxide in the carbon nanotube pores easily [15]. The process using 5 grams of MWCNT is dropped into 375 ml of 20% ammonium sulfate solution and stirred for 3 hours or 4 hours at room temperature. This solution is heated at 110°C with two sulfonation time variables, 3 and 4 hours. The stored particles are filtered, cleaned using distilled water, and used as catalyst supports. Zinc oxide precursors are synthesised from a mixture of zinc acetic and oxalic acid based on loading% ZnO to be used in the pores of MWCNTs. To create a 20% ZnO loading, a 20% oxalic acid solution is used slowly into a 20% acetate zinc solution using titration techniques with impregnation time variations. There are four variations of time impregnation, 6, 8, 10, and 12 hours, respectively. The filtrate obtained is then filtered, washed with distilled water and 10 ml of acetone, and dried at a temperature of 120°C overnight. The final preparation stage is calcination at 500 ° C for 4 hours. The next step is To convert ZnO nanoparticles to the surface of MWCNTS. ZnO and MWCNTS solutions are stirred for 4 hours at room temperature. After mixing, the final solid deposition is filtered and dried overnight at a temperature of 120°C. The catalysts successfully developed in the impregnation of variations with 3 hours sulfonation time were encoded by C6-3, C8-3, C10-3, and C12-3, whereas in the impregnation of variations with 4 hours sulfonation time, encoded by C6-4, C8-4, C10-4, C12-4.

Catalyst characteristics are analysed by testing XRD, BET, and SEM-EDX at the Integrated Laboratory of Diponegoro University (Undip), Semarang City, Central Java. Testing catalyst activity can be done by transesterification with a batch reactor system consisting of a three-neck flask, cooler, hotplate, and magnetic stirrer. The transesterification process by mixing the ZnO/MWCNTS catalyst that has been added by methanol and heated to a temperature of 65 ° C with kesambi oil, which is also heated at a temperature of 65 ° C after stable does the process of calculating the variation of stirring time. There are four variants of time in the transesterification process, ranging from 4 hours to 7 hours by adding 1 hour to each technique and coded CN-04-4J, CN-04-5J, CN-04-6J, CN-04-7J. After the reaction is completed, separate the filtrate and residue; the filtrate will be reheated in the oven at a temperature of 70 ° C for 24 hours; then, the analysis test is carried out.

Kesambi Oil Conversion

The catalyst conversion process is tested to be a conversion of kesambi oil (*Schleicher oleosa*) through a reaction in the batch process. These raw materials are second-class soles that contain several contaminants, such as gum and FFA sap. Before both acid and base reactions are carried out, kesambi oil must be purified to alleviate the latex contained in it with the degumming process as its size. The second step of the degumming process mechanism and the reaction is the same as in previous research without further modification [15]. The biodiesel product then was ready for analysis. The analysis results were used to calculate biodiesel yield as equation (1).

$$\%\text{Yield} = \frac{\text{product mass}}{\text{mass of feedstock}} \text{x}100\% \tag{1}$$

Several parameters determine biodiesel products' quality, density, viscosity, FFA levels, water content, and saponification values. Product yield calculations regarding the number of biodiesel products from density measurements are carried out using this equation.

Otherwise, FFA content was analysed using the titration technique with the base solution. In this work, NaOH solution was used as a base titer, and the FFA content was measured using this formula,

$$\%\text{FFA} = \frac{\text{volume of titer x molarity of NaOH x 28}}{\text{mass of sample}} \text{x}100\% \tag{2}$$

Saponification value (SV) was also measured using a phenolphthalein indicator. HCl was utilised as an acid solution on the titer side, and this value was calculated using the equation,

$$\mathrm{SV} = \frac{\text{volume of titer x molarity of HCl x 56.1}}{\text{sample weight}} \tag{3}$$

RESULTS AND DISCUSSION

Kesambi Oil Characteristics

Kesambi(*Schleichera Oleosa L)* is a tropical forest tree plant one of the Sapindaceae families scattered in the South and Southeast Asian region; the potential oil potential is around 70-73% of dry seeds. Making kesambi oil is expected to be a new source of biodiesel production [3].

Table 1. The content of fatty acids in the oil

Fatty Acid	*Value (%)*	*Fatty Acid*	*Value (%)*
Myristic acid	0.01	*Eicosenoic acid*	28.97
Palmitic acid	7.42	*Eicosadienoic Acid*	0.29
Palmitoleic acid	1.9	*Heneicosanoic Acid*	0.06
Oleic acid	2.59	*Behenic Acid*	1.21
Linolelaidic acid	50.05	*Erucic acid*	1.33
Linoleic acid	5.35	*Lignoceric Acid*	0.02
alpha-Linolenic acid	0.56	*Docosahexaenoic Acid*	0.04
Eicosenoic acid	28.97		

First, kesambi oil was analysed by its FFA content, such as linolelaidat acid, water content, saponification value, viscosity, and density. These characteristics are presented in Table 2, showing that kesambi oil has a high FFA content. But it was lower than kesambi oil used by previous research researchers, reaching 29% WT [16]. The value of this characteristic is very different from the results of other studies because of the authenticity of the harvested crusty[17]. The analysis results in conplanttains fatty acids with carbon structures of double bonds. It contrasts with other natural oils, indicating that kesambi oil consists of high-saturated fatty acid molecules [18].

From the results of measurements, kesambi oil contains high water molecules. It is thought to be caused by the enzymatic conversion of lipase contained in kesambi and secrete water molecules as other additive compounds [19]. Water compounds are a significant limit in deciding the nature of oil. The water molecules contained in oil will cause a fatty substance of hydrolysis into unsaturated fats and glycerin; along these lines, expand the degree of free unsaturated fat. High substances in non-saturated fats will reduce the number of biodiesel products. Because the number of strong acid catalysts is more inhuman than unsaturated fats and solid base catalysts, FFA compounds in kesambi oil are pretty high (15%), which need to be reduced. As a result, kesambi oil must be cleaned before being treated by transesterification; in any case, it causes a reduction in the number of biodiesel. After measuring separation and purification, the purified yield shade was obtained from KO with 0.01% of water compounds.

Table 2. Properties of kesambi oil

KO properties	**Value (%)**
FFA as linolelaidic acid (%wt)	15
Moisture content (%wt)	0.1
Saponification value (mg/gr)	526.61
Viscosity (cSt)	2.14
Density (gr/ml)	0.9074

The structure of unsaturated fat in Kesambi oil has a substance of unsaturated fats roof and 99.8%. Unsaturated fats most consist of linolelaidik acid of 50.05%, eicosenoic acid of 28.97%, and palmitate acid of 7.42%. This composition differs from silitonga et al., which indicates that oleic acid is the most critical component, followed by Arakhidat acid, which is 27.21% [3]. It can happen because the source of kesambi oil is a plant obtained from different plantations, and this causes a diversity of kesambi oil composition [8].

Characteristics Catalyst

ZnO/MWCNTs catalyst is obtained by mixing ZnO deposits and MWCNTs deposits that have been sulfonated. Then the process of testing the characteristics of calcined products in the form of ZnO/MWCNTs catalysts will be analysed with XRD, BET, and SEM-EDX. The XRD test in Figure 1 results in a diffraction pattern of 20% loading ZnO catalyst, which shows the same as ZnO diffraction on JCPDS Card no. 00-036-1451. While MWCNTs still show an amorphous structure. It can be concluded that the existence of MWCNTS does not affect the structure of ZnO crystals [19]. While the bet test is carried out to determine the surface area of the catalyst, the sample tested is pure material MWCNTS, ZnO/MWCNTS catalyst with ZnO loading variables with a ZnO concentration of 20%. MWCNT pure substances have a surface area of 673,242 m2 / g, while ZnO/MWCNT catalysts with a concentration of 20% have a surface area of 157,596 m2 / g.

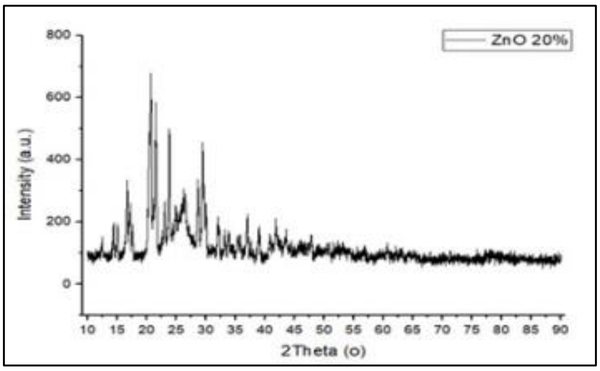

Figure 1. Diffraction pattern catalyst loading ZnO 20%

SEM-EDX test results were used to provide information about the topography, morphology, and composition of the ZnO/MWCNTS catalyst. This can be seen from the pure catalyst sem test in Figure 2 (a) is the result of testing SEM-EDX from pure MWCNTS, which shows the morphology of pure MWCNTS in the form of carbon tubes. Figure (b) shows the results of the SEM-EDX testing from the ZnO/MWCNTS catalyst without adding the the-SO3H functional group. In contrast, figure (c) shows the results of the SEM-EDX testing from the ZnO/MWCNTS catalyst with the addition of the Function Group -SO3H. Figures (B) and (c) show that the morphology of MWCNTS still looks like carbon tubes that indicate no change in shape from MWCNTS, while ZnO can be seen attached to the surface of MWCNTS. It can be concluded that there is no significant change in the effect of ZnO catalyst impregnation and MWCNTS sulfonation on MWCNTS morphology. The composition of this test shows that the catalyst composition is dominated by carbon, at 89.76%.

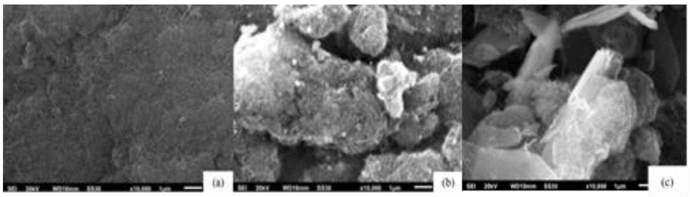

Figure 2. (a) MWCNTs, (b) Morphology of ZnO/MWCNTs without sulfonation, (c) Morphology of sulfonated ZnO/MWCNTs.

Conversion Of Kesambi Oil

The product yield results can be seen in Figure 3; the most significant 3-hour sulfonation time variable was at 12-hour impregnation time with a presentation of 51.59%, while the most negligible product yield was at 6-hour impregnation time of 33.71%. Meanwhile, at the 4-hour sulfonation time, the largest was at 12 hours with a percentage of 52.43%, and the smallest was at 6 hours with a rate of 20.00%.

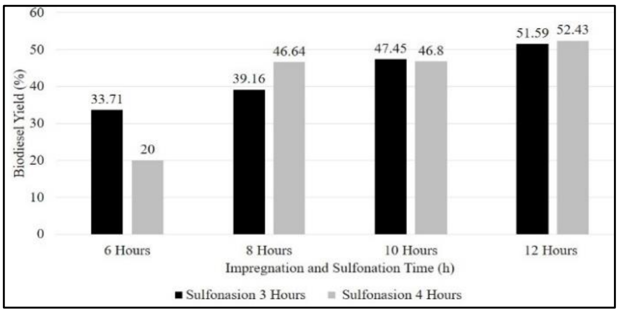

Figure 3**.** The yield of biodiesel product with impregnation and sulfonation time variation.

For this reason, the transesterification time variable uses an impregnation time of 12 hours and a sulfonation time of 4 hours with a transesterification time of 4 hours, 5 hours, 6 hours, and 7 hours. The most significant product result for the stirring time variable in the transesterification process can be seen in figure 4; the best outcome is the time of stirring the transesterification process for 7 hours with a percentage of 85.46%. The lowest product yield 70.95%, was found at 4 hours of reaction time.

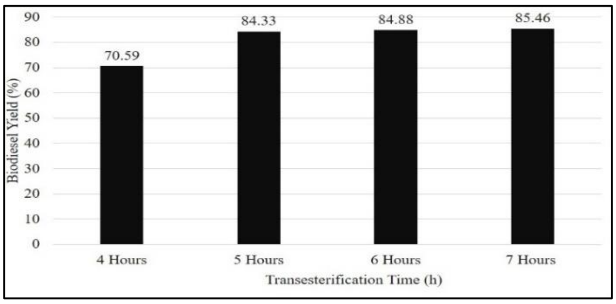

Figure 4. The yield of biodiesel product with transesterification time variation.

The biodiesel results shown in Figure 4 have increased when compared to previous studies with a value below 15% [10]. It can be estimated that the increase in conversion value occurs due to the sulfonation process; the particles owned by ZnO can be dispersed perfectly on the walls of the MWCNTs particle tube. This is different from previous studies that do not use the process of time addition process (h) at the time of sulfonation also affects the results of catalyst conversion; the highest development of catalyst conversion is done with sulfonation 4 hours of 52.43%. The process of testing the catalyst activity by adding time (h) to the transesterification process for 7 hours showed an increase of 85.46%.

CONCLUSION

Based on research data obtained can be concluded:

1. Biodiesel yield from sulfonation time variable 4 hours has a better result than sulfonation 3 hours. While in the best condition, impregnation variables were obtained at 12 hours.
2. The highest biodiesel yield of 85.46% was obtained during a transesterification process of 7 hours

ACKNOWLEDGEMENTS

Thanks submitted to the Ministry of Research, Technology, and Higher Education, who has funded the continuity of this research, and Mrs Prof. Dr Ir. Nyoman Puspa Asri, Ms. who guided in this article's research and writing.

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