Advance Sustainable Science, Engineering and Technology (ASSET)
Vol. 6, No.4, October 2024, pp. 0240404-01 ~ 0240404-10
ISSN: 2715-4211 DOI: https://doi.org/10.26877/asset.v6i4.779

Synthesis of Avocado Seeds Into Biodiesel Using A Catalyst CaO From Blood Cockle Shell

Suprihatin*, Alif Julian Putra Rahmandika, Muhammad Fikri,

Faculty of Engineering and Science, Universitas Pembangunan Nasional "Veteran" Jawa Timur, Jl. Rungkut Madya No.1 Gunung Anyar Surabaya 50241, East Java, Indonesia

*suprihatin.tk@upnjatim.ac.id

Abstract. Biodiesel is an environmentally friendly fuel made from oil vegetables that contain triglycerides. Biodiesel from avocado seed vegetable oil in Indonesian agricultural areas which according to BPS 2022 data, Indonesia produces 183,000 tons of avocados per year. This research aims to gained the effect of adding reaction temperature and Oil : Methanol ratio influence on biodiesel production. The avocado seed oil obtained by soxhlet extraction method, where 50 gram avocado seeds powder extracted with n-hexane solvent in 1 hour extraction time and 60°C temperature giving result 10% yield of avocado seed oil . The CaO catalyst are obtained from Calcination procees of blood cockle shells in 900°C temperature and 4 hours calcination time giving 98.82% CaO Cotent. The biodiesel is produces with 97% methanol reactant and 98.82% CaO catalyst in various methanol volume (30; 40; 50; 60; and 70 ml) and under different temperature conditions (30; 40, 50, 60, and 70 °C). The best result of transesterification process biodiesel is obtained in 50°C and 40ml methanol gets biodiesel yield of 96%, methyl ester content of 99.83%, density of 865 gr/cm^3, viscosity of 2.5 cSt , mgKOH/gr acid number of 0.56 of, and heating value of 9871.6 kcal/kg. Based on the high result of methyl ester content and heating value of biodiesel obtained from the procees, the avocado seed oil biodiesel potentially used as an sustainable energy.

Keywords: Avocado Seed Oil, Blood Cockle Shells, Renewable Energy, Biocatalyst, Biodiesel

(Received 2024-07-03, Accepted 2024-07-27, Available Online by 2024-08-16)

1. Introduction

The impact of the use of fossil energy sources is getting bigger energy needs are increasing and fossil energy reserves are running low. One form of alternative energy that is being developed as a replacement diesel is biodiesel. Biodiesel is a fuel environmentally friendly equivalent to petroleum based diesel and both can be completely mixed without any grouping. In terms of appearance, composition and chemical properties, biodiesel fuel can decompose naturally, has low levels of toxins and emits fewer air pollutants compared to petroleum-based diesel fuel [14]. Biodiesel can be made from vegetable oils that come from natural resources renewable by the transesterification process. Vegetable oil raw materials that can be used in making biodiesel include: palm oil, jatropha, seeds rubber, coconut, soybeans, corn, avocado, sunflower seeds and some other plants [13], [2]. Avocado are one of the products agriculture which is still not utilized optimally as in its part fruit seeds which until now have only been thrown away as waste. Based on data from the Central Statistics Agency (BPS) in 2022, especially in East Java Province produces around 183,000 tons of agricultural products to reduce pollution, use avocado seed waste into vegetable oil.

Research by Dewi 2022 and Redjeki 2021 evaluated avocado seed oil extraction using n-hexane, finding oil yields of 16.66% and 16.54%, respectively, from different extraction volumes and times [5], [8]. Biodiesel production requires a catalyst, and heterogeneous catalysts are preferred for their environmental benefits over homogeneous ones, which are harder to separate from reaction products and can cause pollution. Heterogeneous catalysts like CaO, derived from waste materials such as crab, blood cockle, and snail shells, are more eco-friendly. Research conducted by Saraswati 2023 highlighted the high calcium carbonate content in shellfish waste, which decomposes into CaO during heating and can be used as a catalyst [9]. Research conducted by Azzahro 2021 demonstrated that blood cockle shells can provide a CaO catalyst with 99% purity when heated to 900°C for 4 hours [3]. Studies on biodiesel production from avocado seeds and the use of CaO suggest significant potential. A research conducted by Dwita 2019 achieved 87.9% biodiesel content using avocado seed oil and H2SO4 catalyst, while Azzahro 2021 obtained 78.1% biodiesel from used cooking oil with a CaO catalyst [4], [3]. Avocado seeds, containing fatty acids like linoleic, palmitic, and oleic acids, offer a substantial oil yield (20-30%) and are a viable source for biodiesel due to their high oil content compared to grains and their widespread availability in Indonesia[7], [10].

Biodiesel is generally produced through a transesterification reaction between triglycerides (oils or fats) and simple alcohols (such as methanol, ethanol, propanol, and butanol). Glycerol is simultaneously produced as a byproduct. Stoichiometrically, the transesterification reaction involves 3 moles of alcohol and 1 mole of triglyceride to produce 3 moles of methyl esters (biodiesel) and 1 mole of glycerol. However, due to the reversible nature of the reaction, an excess of alcohol is used in practice to shift the equilibrium toward the desired products to achieve a higher ester yield [12]. The production of biodiesel is influenced by several factors, including the molar ratio of oil to alcohol, reaction temperature, catalyst concentration, stirring speed, and reaction time. A higher molar ratio of oil to alcohol generally results in a greater biodiesel yield, while the reaction temperature must be controlled to avoid methanol evaporation, which reduces yield. Catalyst concentration should be optimized to avoid soap formation that can lower biodiesel yield. Stirring ensures a homogeneous mixture and enhances conversion but can slow the reaction if excessive. Finally, reaction time should be sufficient to allow conversion but not extended beyond equilibrium, as this can decrease the product yield, especially in reversible reactions [11]. The objective of this research is to determine the effect of increasing the transesterification reaction temperature on the methyl ester content produced and the effect of increasing methanol volume on the biodiesel transesterification process. In addition, the quality of the biodiesel produced must meet the SNI 7182:2015 standard.

2. Methods

2.1 Research Method

The materials used in this research are avocado seeds purchased from Fruit Market in Sidoarjo, blood clam shells waster bought from a Fish Market in Surabaya, 97% methanol, n-hexane, and distilled water bought from chemical supplyer in Surabaya.

2.2 Preparation of CaO Catalyst

The biodiesel production process begins with two preliminary steps for preparing the raw materials. The preparation of the CaO catalyst begins with cleaning the blood clam shells using water to remove any adhering dirt and sand. After cleaning, the shells are dried at 105ºC for 1 hour and then coarsely crushed using a mortar and pestle. The crushed shells are calcined at 900ºC for 4 hours [3]. High calcination temperatures are used because CaO begins to form at temperatures above 500ºC, and at temperatures of 900ºC or higher, the CaO that forms will be more active [1]. Therefore, when used as a catalyst, it will be more effective.Once the calcination process is complete, the shells are ground and sieved through a 100 mesh sieve, and then stored in a closed container. To determine the CaO content, an SEM-EDX analysis is conducted on the prepared catalyst.

2.3 Preparation of Avocado Oil

The avocado seeds are first peeled to remove the membrane and washed thoroughly. The seeds are then cut into thin slices using a knife to reduce their size. These slices are dried in an oven at 60ºC for 1 hour. After dryed, the seeds are ground and sieved to a size of 40 mesh. Any oversize particles are re-ground until they meet the 40 mesh specification. The uniformly sized avocado seed powder is subjected to an extraction process. In this process, 150 ml of n-hexane solvent is added and heated to 70ºC. Fifty grams of the powdered avocado seed are placed into a Soxhlet extractor, and extraction is performed for 1 hour [8]. The extracted avocado seed oil is then analyzed by Gas Chromatography Mass Spectometry to determine the Fatty Acid content obtained.

2.4 Biodiesel Transesterification

The process of making biodiesel is carried out by transesterification process, mixing methanol with variables of (30,40,50,60,70 ml) in 50 ml oil at a temperature of (30,40,50,60,70 ºC) for 1 hour at a speed of 250 rpm for 1 hour. After the reaction, the mixture is separated using a separatory funnel; the upper layer is biodiesel, while the lower layer is glycerol. The biodiesel layer is then washed with warm distilled water (50-60ºC) in a 1:1 volume ratio to dissolve any remaining methanol and soap. The mixture is allowed to settle until three distinct layers form: biodiesel at the top, soap in the middle, and water at the bottom. The resulting biodiesel is heated to 105ºC to remove any residual water content. Finally, GC-MS analysis is conducted to assess the quality of the biodiesel and the extent of oil conversion to biodiesel.

2.5 Data Analysis

The methyl ester content and methyl ester composition were analyzed using Gas Chromatography–Mass Spectrometry (GC-MS) method, then the CaO content and atom compostion were analyzed using SEM-EDX method. The other parameters such as the density, viscosity, acid number and yield were analyzed using basic laboratory analysis method.

3. Results and Discussion

3.1 Results of Avocado Seed Oil Extraction

After conducting research, The analysis results revealed the oil yield and its fatty acid content. The content of fatty acid was tested at the Laboratory of the Institute for Industrial Research and Consultation, Ketintang, Surabaya, was as follows:

Table 1. Yield Result of Avocado Seed Oil

Analysis Parameter	**Yield**
Essential Oil	10%

Table 2. Fatty Acid Analysis Resulf of Avocado Seed Oil

Fatty Acid Composition	**Content (%)**
Palmetic Acid	14,8
Stearic Acid	2,4
Oleic Acid	68,3
Linoleic Acid	9,5

Based on the measurements, the yield of essential oil obtained from the extraction process of avocado seeds was 10% from 50 grams of powdered avocado seeds. The analysis results show a discrepancy according to the theory [5] that the amount of vegetable oil that can be extracted from avocado seeds is quite high, between 20-30%, whereas the extraction of avocado oil using n-hexane solvent only yielded about 10%. This can be influenced by several factors according to research [18], including utilizing the boiling point of the solvent to obtain a high extract oil yield based on temperature and extraction time. Temperature and time factors greatly influence the extract yield. The free fatty acid content is significantly affected by these two factors to obtain good quality oil. Additionally, the type of avocado seed used depends on the condition of the avocado seed as it can affect the oil content contained in the seed.

3.2 Results of CaO Catalyst

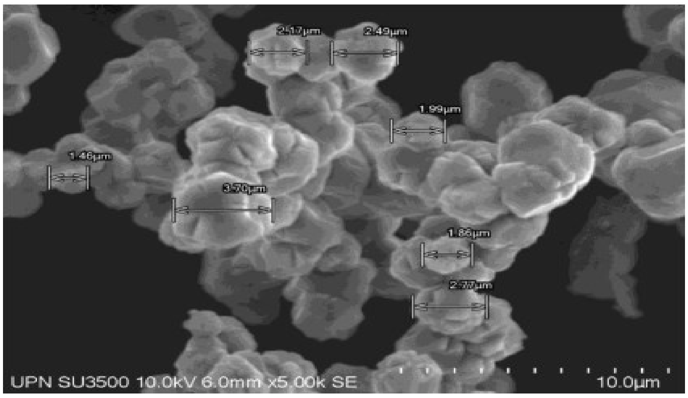

Figure 1. SEM Results of Blood Clam Shells at 5000x Magnification

Tabel 3. Analytical Result of CaO Catalyst Content

Atom Element	Content (%)
Ca	56,43
O	42,39
C	1,18

The CaO catalyst analyzed using SEM-EDX at the Instrumentation Laboratory, LPT UPN "Veteran" Jawa Timur, showed that the morphology and element content contained in the CaO catalyst from blood cockle shell waste showed a calcium content of 56.43%, oxygen content of 42.39% and carbon content of 1.18%. Based on the results of the CaO catalyst analysis, the CaO morphology was spherical and the CaO content was 98.82%. The surface of the CaO catalyst has an irregular shape and bonds together to form large aggregates. Based on Indonesian National Standards (SNI), the minimum CaO level is appropriate, namely more than 98%.

The analysis results of the catalyst are consistent with the theory [19] that the morphology of the obtained calcium oxide catalyst has an irregular shape, with some of them bonded together to form large aggregates. The synthesis of the CaO catalyst is carried out to enhance the catalytic ability of a material, aiming to improve its catalytic selectivity and stability. The surface properties of a material have a significant impact on its applications. The crystallinity of the material's structure will affect the surface properties of the material. The better the crystallinity of the material's structure, the better the surface properties of the material. Additionally, determining the surface area and surface morphology of a material also influences its surface properties. The small size of catalyst particles can increase the surface area of the catalyst, thereby enhancing the contact area between the active phase of the catalyst and the reactants in the transesterification process [17].

3.3 Characteristics of Biodiesel

The extracted avocado seed oil, amounting to 50 ml, was transesterified using 2 grams of CaO catalyst with varying volumes of methanol (30; 40; 50; 60; and 70 ml) under different temperature conditions (30; 40, 50, 60, and 70 oC) at a stirring speed of 250 rpm for 1 hour. The research results are as follows:

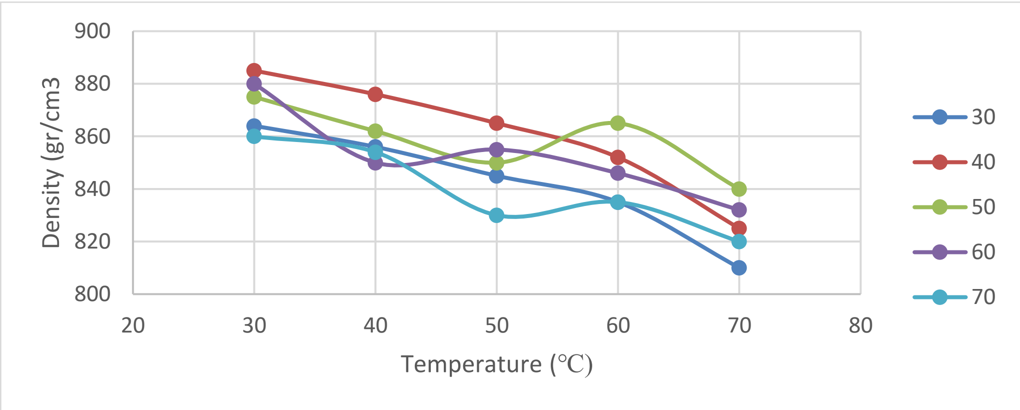

Figure 2. Relationship Between Reaction Temperature, Methanol Volume and Density

The density of biodiesel is influenced by reaction temperature and methanol volume. At 30°C, high density indicates incomplete conversion of fatty acids. As the temperature rises to 40-70°C, density decreases significantly due to better conversion. Higher temperatures increase molecular collisions, enhancing methyl ester formation. More methanol also leads to higher conversion rates, reducing the impact of fatty acids on density. However, discrepancies in density results may occur due to inadequate washing, leaving impurities like glycerol. The best density of 850 g/cm³ was achieved at 50°C with 50 ml of methanol.

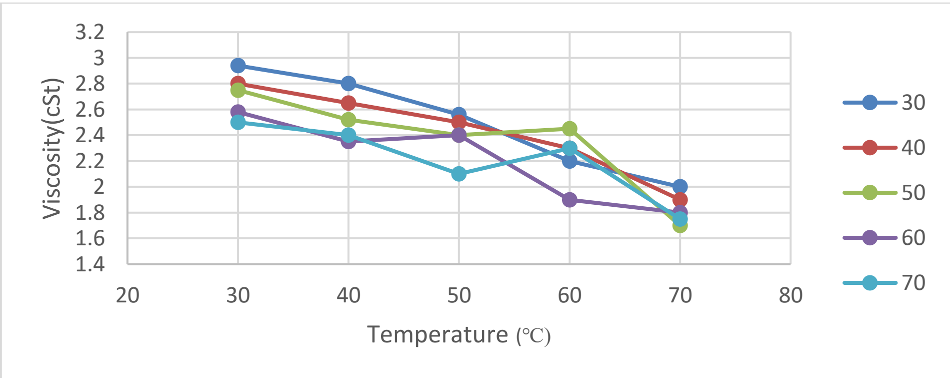

Figure 3. Relationship Between Reaction Temperature, Methanol Volume and Viscosity

The viscosity of biodiesel is influenced by reaction temperature and methanol volume. Higher reaction temperatures and increased methanol ratios reduce viscosity by converting more fatty acids to methyl esters. Conversely, lower temperatures and lower methanol ratios result in higher viscosity.

Discrepancies in viscosity results are due to inadequate washing, leaving glycerol impurities. The optimal biodiesel viscosity of 2.3 cSt was achieved with 40 ml of methanol at 60°C. The test results show that the viscosity value obtained is higher compared to conventional diesel. In the context of fuel, viscosity affects the injection and atomization process of the fuel inside the engine cylinder [16]. Additionally, higher viscosity can lead to less optimal fuel distribution, reduced combustion efficiency, and increased emissions.

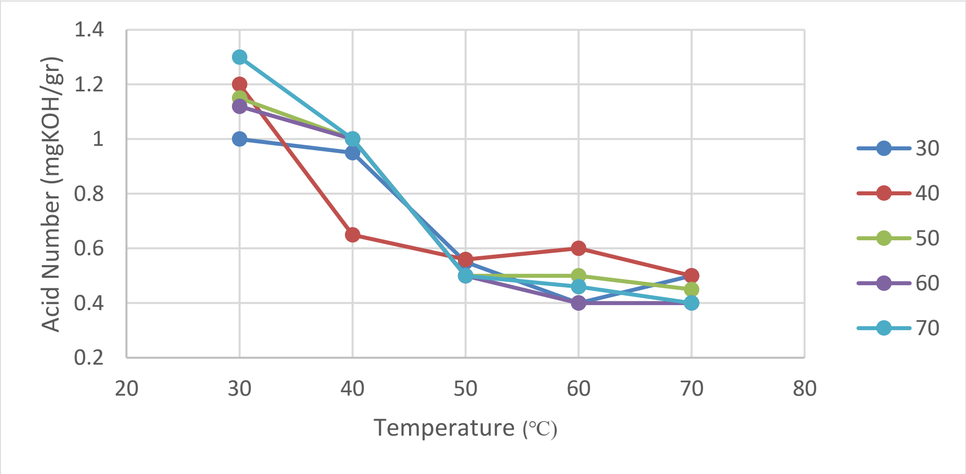

Figure 4. Relationship Between Reaction Temperature, Methanol Volume and Acid Number

The acid number of biodiesel, which indicates the amount of free fatty acids, is influenced by reaction temperature and methanol volume. A lower acid number signifies better biodiesel quality due to more complete conversion of fatty acids to methyl esters, while a higher acid number indicates incomplete conversion. High acid numbers are caused by insufficient methanol ratios and inadequate washing, leaving impurities and free fatty acids. The optimal acid number of 0.4 mg KOH/g was achieved with 70 ml of methanol at 70°C, resulting in approximately 98.87% methyl ester content and a lower calorific value.

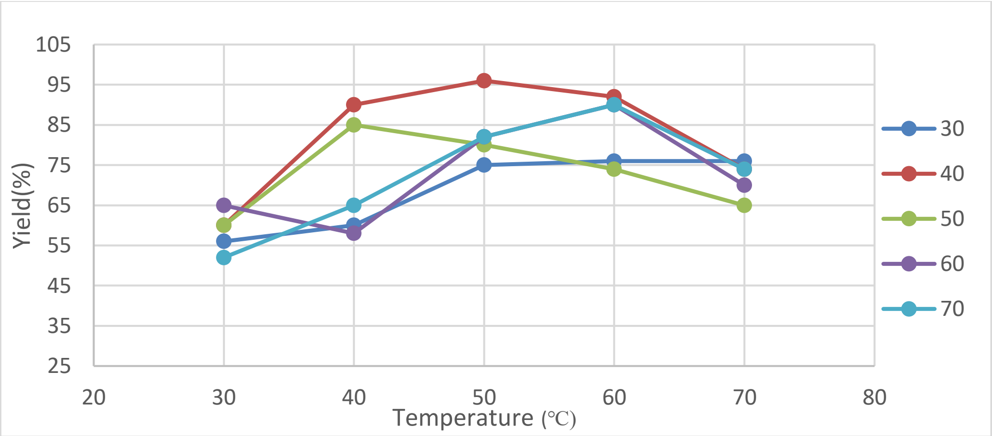

Figure 5. Relationship Between Reaction Temperature, Methanol Volume and Yield

The percentage yield of biodiesel is influenced by reaction temperature and methanol volume. Higher temperatures accelerate the transesterification process, enhancing the formation of methyl esters from avocado seed oil and methanol. Increasing methanol volume also increases yield by promoting more extensive conversion. The best yield of 96% was achieved with 40 ml of methanol at 50°C, emphasizing the critical role of these factors in maximizing biodiesel production efficiency.

3.4 Analysis of Gas Chromatography–Mass Spectrometry

Gas Chromatography–Mass Spectrometry (GC-MS) testing was conducted on the sample with the highest yield value. According to theory, the higher the yield value, the greater the amount of triglycerides converted into biodiesel. The tested sample was among the best results in terms of yield obtained. However, its acid value did not meet the SNI 7182:2015 standard. Based on the GC-MS analysis, the sample contained methyl ester content that meets the SNI standard, although its acid value parameter did not comply with SNI. This proves that the GC-MS test results are consistent with the theory, indicating that the greater the yield obtained, the greater the conversion of triglycerides into biodiesel. The methyl ester biodiesel content analyzed by GC-MS will be presented in the figure below.

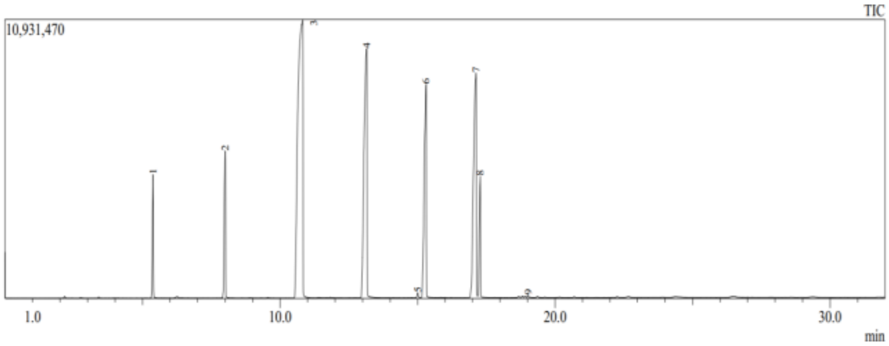

Figure 6. Gas Chromatography of biodiesel product with 40 ml methanol at 40°C

Table 3. Results of Mass Spectrometry analysis of methyl ester composition at 40 ml methanol and 40°C.

Methyl Ester Component	Content (%)
Methyl laurate	36,05
Methyl myristate	18,35
Methyl oleate	16,22
Methyl linoleate	11,47
Methyl palmitate	7,28
Methyl decanoate	6,19
Methyl stearate	3,46

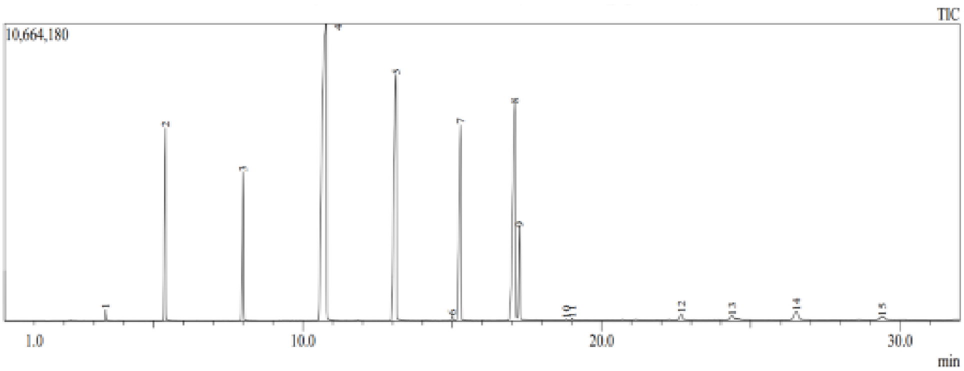

Figure 7. Gas Chromatography of biodiesel product with 40 ml methanol at 50°C

Table 4. Results of Mass Spectrometry analysis of methyl ester composition at 40 ml methanol and 50°C.

Methyl Ester Component	Content (%)
Methyl laurate	37,9
Methyl myristate	19,81
Methyl oleate	17,57
Methyl Palmitate	12,53
Methyl decanoate	5
Methyl stearate	3,86
Methyl octanoate	3,1

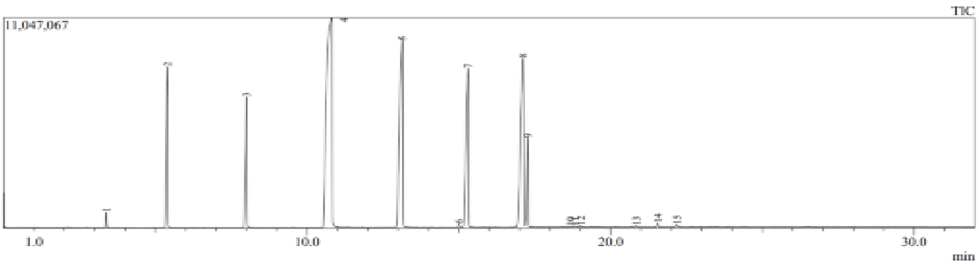

Figure 8. Gas Chromatography of biodiesel product with 40 ml methanol at 60°C

Table 5. Results of Mass Spectrometry analysis of methyl ester composition at 40 ml methanol and 60°C.

Methyl Ester Component	Content (%)
Methyl laurate	37,37
Methyl Miristate	17,83
Metil Oleat	16,28
Methyl palmitate	10,58
Methyl decanoate	6,59
Methyl stearate	5,38
Methyl octanoate	3,06

Based on the results of GCMS analysis, it shows the results of chromatography gas from 40 ml variables at temperatures of 40oC, 50oC and 60oC. In the graphic image, several peaks can be seen containing methyl ester compounds. Based on the table, the results of mass spectometry analysis show that the compounds that make up the methyl esters in this variable consist of two groups, namely saturated fatty acid methyl esters: methyl laurate, methyl myristate, methyl palmitate, methyl decanoate, and methyl stearate, and methyl octanoate. Unsaturated fatty acid methyl esters: methyl oleate and methyl linoleate with a total methyl ester content in the 40 ml variable at a temperature of 40oC 99.02%, in the 40 ml variable methanol at a temperature of 50oC 99.83%, and in the 40 ml variable at a temperature of 60 oC 97.09%.

3.4 Analysis of Calorific value

Table 6. Results of Analysis of the Calorific Value of Biodiesel

Variable (Volume Methanol : Temperature)	Calorific Value (Kkal/kg)
40 ml : 40 oC	9426,5
40 ml : 50 oC	9871,6
40 ml : 60 oC	16,28

Based on table 7, the results of the analysis of the calorific value of biodiesel in the variable volume of 40 ml methanol at a temperature of 40-60 oC/ The calorific value results obtained have met the standard (SNI 7182-2015) with a calorific value range of 10160 - 11000 Kcal/kg. Based on the results obtained, it can be concluded that the greater the reaction temperature used, the greater the calorific value produced. With the high reaction temperature, more and more of the fatty acid components contained in avocado seed oil are converted into biodiesel products, so the density decreases. The low density value of biodiesel causes the resulting calorific value to be higher. Based on GC-Ms analysis at a variable temperature of 40oC, it has a greater composition of unsaturated (double) fatty acids compared to variable temperatures of 50oC and 60oC. The calorific value of fuel determines the amount of fuel consumed per unit time. The higher the calorific value of a fuel, the more efficient the energy produced will be because it produces more heat with less mass. The calorific value represents the heat energy released during the complete combustion of a unit mass of fuel. The calorific value or highest heating value (HHV) is an important property that determines the energy content and the clean and efficient energy use of a fuel. Therefore, biomass energy sources have several advantages, not only as renewable energy but also having better combustion results compared to conventional diesel [20].

The characteristics of biodiesel aim to identify and determine the properties of a material compared to the established biodiesel quality standards. In this study, various tests were conducted including acid value, density, viscosity, calorific value, methyl ester content (FAME), and yield. Compared to the research by [15], this study has relatively lower viscosity and calorific value but still meets the SNI standards. The acid number in that study was 0.4, which meets the SNI standards, different from this study which has an average acid number of 0.4 - 1.3 cSt. The methyl ester content in this study is higher compared to that research, at 94.05%.

4. Conclusion

biodiesel from the synthesis of avocado seeds using a CaO catalyst from blood cockle shells produced the largest yield of 96% with a methyl ester content of 99.82%. The best temperature and addition of methanol are obtained at a temperature of 50 oC and 40 ml of methanol. The transesterification result is biodiesel or methyl ester, while the by-product is glycerol. Further analysis shows that the relationship between the molar ratio of methanol and variations in transesterification temperature is directly proportional to the biodiesel yield. This indicates that the higher the temperature and molar ratio, the better the biodiesel produced. However, at a temperature variable of 70°C and with a methanol volume of 60 ml, there is a significant decrease, indicating that the reaction is shifting towards the by-product, glycerol. Based on the characterization of the biodiesel conducted, it has better characteristics compared to conventional diesel. Further research is needed on the optimization of biodiesel production using various materials and catalysts to obtain more efficient biodiesel as a replacement for conventional diesel.

Acknowledgements

Thank you from the author to the parties who helped carry out this research, especially to the UPN "Veteran" East Java research laboratory as well.

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