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Production of Biodiesel from Candlenut Seed Oil (*Aleurites Moluccana* Wild) Using a NaOH/CaO/Ca Catalyst with Microwave Heating

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Abstract

Oil fuels derived from fossils are non-renewable, so over time, they will run out and have a negative impact on air pollution. To overcome this, there is a need for environmentally friendly alternative fuels from renewable sources such as biodiesel. This research used microwave heating with a CaO catalyst. NaOH-impregnated snail shells and active carbon support. This research aims to determine the effect of power on the conversion of candlenut seed oil into biodiesel using the NaOH/CaO/CA catalyst both in terms of compliance with the SNI 7182-2015 standard and analysis using GC-MS (Gas Chromatography-Mass Spectrometry). The synthesis of the NaOH/CaO/CA catalyst was carried out through wet impregnation and calcination at a temperature of 500°C and analyzed using gas sorption analysis (GSA). Then proceed to the transesterification process, where the power for microwave heating was varied to 300, 450, and 600 watts with a mole ratio of esterified oil and methanol, namely 1:10 for 3 minutes. The analysis results of the NaOH/CaO/CA catalyst using the GSA instrument have a surface area of 9.306 m^2/g, pore volume of 0.033 cc/g, and pore diameter of 14.043 nm. Meanwhile, the results of the biodiesel analysis showed that the optimum biodiesel yield was 85.625% at 600 watts of power and had a kinematic density and viscosity that met the SNI 7182-2015 biodiesel standards. Analysis of biodiesel characteristics using GC-MS showed that the three most optimum biodiesel components were hexadecanoic acid, methyl ester (22.664%), 9,12-Octadedecadienoic acid (Z,Z)-, methyl ester (30.176%) and 9-Octadecenoic acid (Z)-, methyl ester (38.656%).

1. Introduction

BPS-Statistics Indonesia (BPS) data shows that the number of motorized vehicles in North Sumatra province increases yearly [1]. Oil fuels that come from fossils are non-renewable, so they will eventually run out and negatively impact air pollution. Therefore, to overcome this problem, there is a need for alternative fuel from renewable sources such as biodiesel [2].

Biodiesel is an alternative fuel that can be produced continuously because it comes from vegetable oils or animal fats and involves alcohol through a chemical process. Biodiesel is renewable and environmentally friendly because it has lower carbon monoxide than fossil fuels. In general, biodiesel uses raw materials in the form of edible oil [3]. However, the use of edible oil has a weakness, namely competition with human consumption, which can increase biodiesel production costs. To overcome this weakness, developing oil with non-food properties is necessary [4]. One of the raw materials for oil that has non-edible oil properties comes from candlenut seed oil [5].

In candlenut oil (*Aleurites moluccana Wild*), there is α- eleostearic acid, a toxic compound, making candlenut oil a non-food in nature [6]. The oil content in candlenut seeds is relatively high, around 55-66% of the total weight of candlenut seeds. One use of candlenut oil is as fuel for oil lamps because it is flammable [7]. The raw material for candlenut seed oil has a high free fatty acid content of around 5.4%, so to be able to use it in the transesterification process, it must go through an esterification process to reduce the free fatty acid content to ≤2% using a sulfuric acid catalyst [8]. Meanwhile, biodiesel is formed using homogeneous and heterogeneous base catalysts in the transesterification process. Heterogeneous catalysts have many advantages, including easier separation of product and catalyst, reusability, and reduced amounts of wastewater generated [9]. One of the heterogeneous base catalysts is the calcium oxide (CaO) catalyst.

The snail shell is the unused part of the snail's body. Snail shells contain calcium (calcite; $CaCO_3$) with levels of 88.54%. So, it is very suitable as a raw material source for making heterogeneous base catalysts, namely calcium oxide (CaO) catalysts [2]. There are weaknesses in using the CaO catalyst as a heterogeneous base catalyst, namely that it has a low surface area and CaO and glycerol are difficult to separate. To overcome this problem, wet impregnation of NaOH, an alkali metal, was carried out in this research. As well as inserting support such as activated carbon [10].

Biodiesel production conventionally employs hotplate heating, a method that is known to be time-consuming, requiring hours for completion. Therefore, a modern heating method, such as microwave technology, can be a solution to expedite the biodiesel production process. Beyond the time efficiency gained, microwave heating offers additional benefits, including reduced energy consumption, enhanced yield, and the potential to minimize the reliance on alcohol solvents [4].

This research aims to determine the effect of microwave power and NaOH/CaO/CA catalysts in converting candlenut seed oil into biodiesel (fatty acid methyl ester). The uniqueness of this project is the use of candlenut oil as a biodiesel source. Candlenut oil is an inedible product. It cannot be used in food because it is poisonous. Candlenut oil is one of the mainstay agricultural products of Karo Regency. Therefore, this research is anticipated to contribute to the elevation of the economic value of candlenut oil. Apart from that, the catalyst used in this project is also a renewable catalyst and is categorized as a heterogeneous catalyst, which generally uses a homogeneous catalyst in making biodiesel. Homogeneous catalysts are difficult to remove once biodiesel is formed [11]. Therefore, CaO from snail shells is a promising catalyst. Another important aspect of this project is the use of microwave heating.

2. Experimental

2.1. Materials

The materials used in this research were 200 g of directly processed candlenut seed oil, distilled water, solid sodium hydroxide (NaOH) (Merck, 99%), solid potassium hydroxide (KOH) (Merck), 1000 g of a snail shell, methanol (Merck, 99.99%), ethanol (Merck, 96%), phenolphthalein (PP) indicator, concentrated sulfuric acid (H_2SO_4), anhydrous sodium sulfate (Na_2SO_4), sodium bicarbonate ($NaHCO_3$), acetic acid (CH_3COOH), hydrochloric acid (HCl), and benzoic acid ($C_7H_6O_2$).

2.2. Catalyst Preparation

The field snail shells were cleaned with water and placed in the oven for 10 hours at 105°C. Next, the shells were crushed using a mortar and pestle, then blended to soften and sift using an 80 mesh sieve. Then, the powdered shells were calcined using a furnace at a temperature of 900°C for 4 hours [9]. Twenty-one g of CaO and 9 g of activated carbon (CA) with a mass ratio of CaO to CA of 7:3 were weighed. NaOH 30% w/w (10.956 g) was added to 100 mL of distilled water in the Erlenmeyer flask, which already contained CaO and CA. The mixture was stirred for 1 hour and left overnight at room temperature. Subsequently, it was dried in the oven at 100°C for 5 hours. The next stage involved calcination at 500°C for 5 hours. Once completed, the catalysts were cooled in a desiccator [12].

2.3. Catalyst Characterization

The CaO catalyst from the field snail shell that had been produced was analyzed using XRD to determine the type of mineral formed. Moreover, the NaOH/CaO/CA catalyst was analyzed using the gas sorption analyzer (GSA) instrument that implemented Brunauer-Teller (BET) and Baret Joiner Halenda (BJH) methods to determine the surface area, pore volume, and pore diameter.

2.4. Determination of %FFA (Free Fatty Acid) of Candlenut Kernel Oil

A quantity of 2.043 g of processed candlenut oil was immediately dissolved in 25 mL of methanol and then refluxed to boil for 10 minutes. The reflux results were added with three drops of phenolphthalein indicator solution and titrated with 0.1 N KOH solution until a pink-colored mixture was obtained [13]. %FFA was calculated using Equation (1).

$$\%\text{FFA} = \frac{V_{KOH} \times N_{KOH} \times \text{Molecular weight of free fatty acids}}{\text{Sample mass} \times 1000} \times 100\% \quad (1)$$

2.5. Biodiesel Production

2.5.1. Esterification Reaction

Candlenut kernel oil (110 g) was placed in a three-neck round bottom flask and subjected to microwave irradiation. The esterification reaction was initiated by reacting the oil with methanol in the presence of concentrated H_2SO_4 as a catalyst. The mole ratio of oil to methanol was maintained at 1:12, with the catalyst content set at 5% (w/w) relative to the weight of the oil. Stirring was facilitated using a mixer, and the system was heated at 450 watts of power for 5 minutes. Following the esterification reaction, the resulting mixture was transferred to a separating funnel and allowed to stand until two distinct phases formed. The lower layer contained impurities, constituting a mixture of H_2SO_4, water, and residual methanol.

Meanwhile, the upper layer comprised a mixture of esters and oil. The upper layer was extracted and washed with hot distilled water at 60°C. Subsequently, the washed mixture was heated in an oven at 105°C for 30 minutes to reduce the water content [14]. The subsequent step involved the determination of the %FFA resulting from the esterification process.

2.5.2. Transesterification Reaction

A quantity of 25.01 g of oil obtained from the esterification process was introduced into a three-neck boiling flask. A CaO/NaOH/CA catalyst, constituting 3% (w/w) of the total weight of oil from esterification, was blended with methanol at a mole ratio of oil to methanol set at 1:10. The reflux device series was carried out in a microwave where the microwave will function as a heating device as per the reference from the research [15]. Subsequently, power was set at variations of 300 watts (medium), 450 watts (medium-high), and 600 watts (high), with a stirring duration of 3 minutes using a mixer connected to the microwave shown in Figure 1.

The resulting transesterification product was centrifuged at a speed of 2000 rpm for 10 minutes, leading to the formation of two distinct layers: the upper layer comprised biodiesel, while the bottom layer contained glycerol. The two layers were then separated, with the biodiesel (top layer) being extracted and washed with hot distilled water at 60°C. Subsequently, the biodiesel was heated in an oven at 105°C for 30 minutes. The resulting product was then weighed, and the %yield was calculated using Equation (2) [16]. The catalyst was already in a separate phase from the biodiesel. In this case, the catalyst was at the bottom of the reaction vessel. Once the biodiesel was removed, the catalyst was left in the flask.

$$\%\text{Yield} = \frac{W_{biodiesel}\,(g)}{W_{Candlenut\ kernel\ oil}\,(g)} \times 100\% \qquad (2)$$

2.6. Biodiesel Characterization

2.6.1. Biodiesel Density

The pycnometer was cleaned and dried, and its empty weight was measured. Subsequently, samples were placed into the pycnometer and sealed. The absence of bubbles in the pycnometer was observed. The pycnometer containing the sample was then reweighed, and the density was calculated based on the measurements obtained using Equation (3) [17].

$$\rho_{biodiesel} = \frac{W_{(pycnometer+biodiesel)} - W_{(pycnometer\ empty)}}{V_{pycnometer}} \qquad (3)$$

Figure 1. Reflux circuit with microwave heating

2.6.2. Biodiesel Viscosity

Viscosity testing was carried out at a temperature of 40°C using an Ostwald Viscometer (SI Analytics, 509 03) to measure the time required for the sample to pass the specified limit. To determine the kinematic viscosity of a liquid sample, apart from the flow time, data regarding the density of the sample were required for fluid [18]. Biodiesel viscosity ($\eta_{biodiesel}$) was calculated using Equation (4).

$$\eta_{biodiesel} = \frac{\rho_{biodiesel} \times t_{biodiesel}}{\eta_{distilled\ water} \times t_{distilled\ water}} \times \eta_{distilled\ water} \qquad (4)$$

2.6.3. Biodiesel Characterization Analysis using GC-MS

Biodiesel was characterized using GC-MS to determine biodiesel components and the levels of methyl ester fatty acids formed.

3. Results and Discussion

3.1. Catalyst Preparation

The calcium oxide (CaO) powder exhibits a white color with a subtle hint of grey. Following the sieving process, the field snail shell powder weighed 550.114 g, and subsequent calcination reduced the powder's weight to 340.133 g. The decomposition reaction of $CaCO_3$ into CaO through the calcination process can be written as in Equation (5).

$$CaCO_{3(s)} \rightarrow CaO_{(s)} + CO_{2(g)} \qquad (5)$$

In this study, NaOH served as the metal, and activated carbon was the support material. The addition of supporting materials aims to enhance the surface area, allowing for the subsequent incorporation of NaOH or other embedded substances to increase catalyst activity. The NaOH/CaO/CA catalyst formed was 35.908 g and exhibited a white-brown color.

3.2. Catalyst Characterization

Calcium oxide (CaO) underwent X-ray diffraction (XRD) analysis to qualitatively identify the components formed in catalyst formation by examining the diffraction peaks generated. The diffractogram in Figure 2 reveals several dominant peaks, characteristic of CaO, positioned at 2θ = 32.19°, 37.34°, 53.85°, 64.14°, and 67.36°, aligning with the CaO standard (ICDD No.01-070-4068). In addition to the CaO peaks, another set of peaks indicative of $CaCO_3$ and $Ca(OH)_2$ characteristics is evident at 2θ = 29.39°, 39.39°, 47.38°, 47.56°, and 48.49°, and 2θ = 28.71°, 34.09°, 34.14°, 47.03°, 47.19°, and 50.81°.

Notably, the $CaCO_3$ and $Ca(OH)_2$ diffraction peaks exhibit relatively lower intensities compared to the pronounced intensity of the CaO diffraction peaks. The presence of $CaCO_3$ and $Ca(OH)_2$ compounds in the calcined shell powder suggests potential reactions of CaO with air or water vapor during the process [19]. Intensity in the pattern XRD diffraction is related to the level of crystallinity, where generally, the higher the crystallinity, the greater the particle size.

Table 1. NaOH/CaO/CA catalyst analysis results using BET and BJH methods

Catalyst	Surface area (m^2/g)	Pore volume (cc/g)	Average pore diameter (nm)	Reference
CaO	7.0	0.0312	14.8	[20]
NaOH/CaO/CA	9.306	0.033	14.043	This research

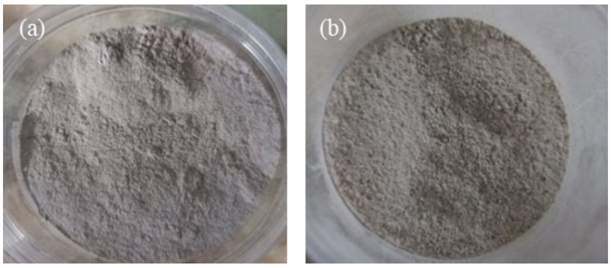

Figure 2. (a) CaO catalyst and (b) NaOH/CaO/CA catalyst

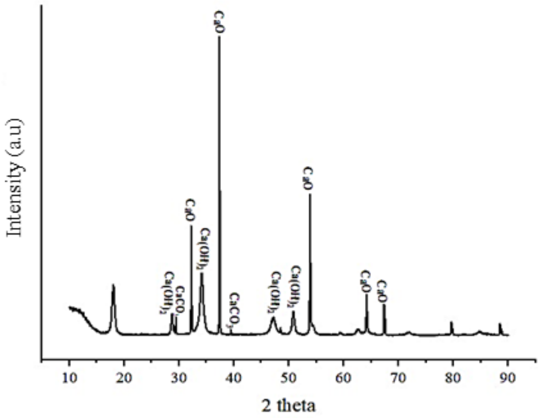

Figure 3. XRD diffractogram of a spotted snail (field snail) shell after calcination

Therefore, in this study, the intensity results of the CaO compound were classified as high, which indicates that CaO is a crystalline phase and the particles are relatively large in size [21]. Apart from these three compounds, there are compounds or impurities which are indicated by the angle 2θ = 19. Based on research by Ismida and Bahri [22], in addition to being primarily composed of the major component, $CaCO_3$, there are also other components present in relatively lower quantities, such as aluminum oxide (alumina). This compound was identified in the diffractogram of calcined snail shell powder with an angle of 2θ = 19.90. However, these alumina compounds are characterized by low intensity [23].

The NaOH/CaO/CA catalyst was characterized using gas sorption analysis (GSA) to determine the surface area, total pore volume, and average pore diameter on the effect of the addition of NaOH metal and activated carbon (support) using the BET and BJH methods.

Table 2. Biodiesel yield from power 300, 450, 600 watts

Power variation (watt)	Yield (%)
300	63.482
450	70.014
600	85.625

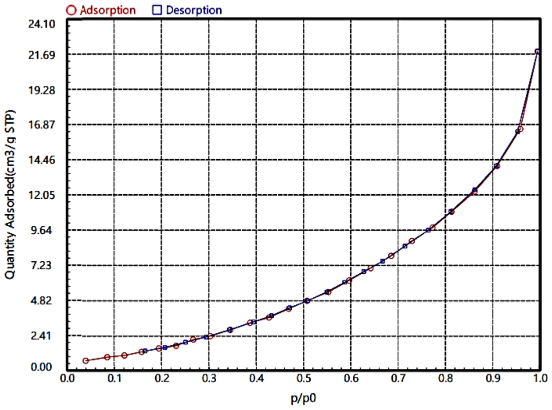

Figure 4. N_2 gas adsorption-desorption isotherm curve on NaOH/CaO/CA catalyst

Based on Table 1, data obtained from the surface area of the NaOH/CaO/CA catalyst is 9.306 m^2/g, which means it has increased compared to the surface area of a single CaO catalyst of 7 m^2/g. Meanwhile, the pore volume and pore diameter values are not much different from single CaO. This is thought to be due to the lack of distribution of Na^+ metal on the CaO catalyst.

The isotherm curve in Figure 3 exhibits type III characteristics, indicating lateral interactions between the adsorbed molecules that are stronger than the interaction between the surface of the adsorbent and the adsorbate [24]. In accordance with the International Union of Pure and Applied Chemistry (IUPAC) classification, porous materials are categorized into three classes: microporous materials (pore diameter < 2 nm), mesoporous materials (pore diameter 2-50 nm), and macroporous materials (> 50 nm). Consequently, the catalyst produced in this research is identified as a mesoporous material, specifically with a pore diameter of 14.043 nm [25].

3.3. Biodiesel Production

The obtained candlenut seed oil exhibited an FFA content of 4.51%, indicating a relatively high level for the transesterification process. Elevated FFA values during transesterification can lead to undesired saponification (soap formation). In order to mitigate this problem, an esterification process was conducted to reduce the FFA content of the candlenut seed oil. The resulting esterification product demonstrated an FFA content of 2.189%, enabling the progression to the subsequent transesterification process. The transesterification yielded two distinct phases. The upper phase consisted of clear yellow fatty acid methyl ester (biodiesel), while the lower phase comprised yellowish-brown glycerol. Different microwave power settings, such as 300, 450, and 600 watts, were employed during the transesterification process. The biodiesel yield results for each power setting are detailed in Table 2.

Table 3. Composition of 600-watt biodiesel (FAME content) using GC-MS analysis

Retention time	Empirical formula	Compound	Area	Relative composition (%)
9.490	$C_{15}H_{30}O_2$	Methyl tetradecanoate (methyl myristate)	1640756.61	0.405
10.920	$C_{17}H_{34}O_2$	Hexadecanoic acid, methyl ester (methyl palmitate)	91801299.83	22.664
12.051	$C_{19}H_{34}O_2$	9,12-Octadecadienoic acid (Z,Z)-,methyl ester (methyl linoleic)	122225418.05	30.176
12.097	$C_{19}H_{36}O_2$	9-Octadecenoic acid (Z)-, methyl ester (methyl oleic)	156574339.33	38.656
12.224	$C_{19}H_{38}O_2$	Methyl stearate (methyl stearate)	15882847.34	3.921
13.643	$C_{21}H_{40}O_2$	Cis methyl 11-eicosenoate	1641909.73	0.405
34.593	$C_{19}H_{36}O_2$	14-Octadecenoic acid, methyl ester (methyl oleic)	6085925.03	1.503

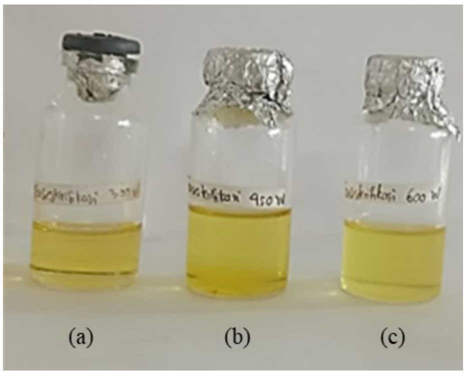

Figure 5. Transesterification process using microwave heating for biodiesel production at different power levels of (a) 300 watts, (b) 450 watts, and (c) 600 watts

According to Table 2, the most optimal power level for biodiesel production is 600 watts, resulting in a yield of 85.625%. Temperature and power are interrelated; higher power levels correspond to elevated temperatures. In alignment with Arrhenius' principle, which highlights temperature as a critical factor influencing reaction speed (k), an increase in temperature is expected to enhance reaction kinetics, leading to more favorable outcomes [26]. Therefore, the observed optimal results at 600 watts align with the concept that higher temperatures can positively impact transesterification. The transesterification process, conducted using microwave heating, is visually represented in Figure 5.

The production of biodiesel necessitates energy, and in this study, microwave power was employed for this purpose, where 1 J/s is equivalent to 1 watt. The energy consumption to produce 1 gram of biodiesel is as follows: 600 watts of power requires 4.93 Kj, 450 watts of power necessitates 4.52 Kj, and 300 watts of power demands 3.33 Kj.

3.4. Biodiesel Characterization

3.4.1. Biodiesel Density

The density of the biodiesel produced in this study falls within the range of 850-890 kg/m^3, indicating compliance with the SNI 7182-2015 biodiesel standard. Notably, there is a slight difference in the density of biodiesel produced at different power variations. Specifically, the density value for biodiesel produced at 600 watts is measured at 879 kg/m^3. The observed decrease in density at 600 watts can be attributed to the increased power, leading to elevated temperatures that potentially accelerate the reaction kinetics. Consequently, larger methyl esters may be formed, contributing to the density variation observed [27].

3.4.2. Biodiesel Viscosity

The viscosity values obtained for each microwave power variation in this study fall within the specified range of the SNI 7182-2015 biodiesel standard, which is 2.3-6.0 cSt. Remarkably, the biodiesel produced at 600 watts exhibits the lowest viscosity, measuring 3.51 cSt, and aligns most closely with the standard requirements compared to other biodiesel samples. The elevated kinematic viscosity observed in the biodiesel produced at 300 watts is likely influenced by the presence of a long carbon chain and high fatty acid saturation, suggesting that the conversion to biodiesel might not be complete at this power level. This could be attributed to the insufficient temperature during the 300-watt, 3- minute process, leading to incomplete conversion and temperature inconsistencies [28].

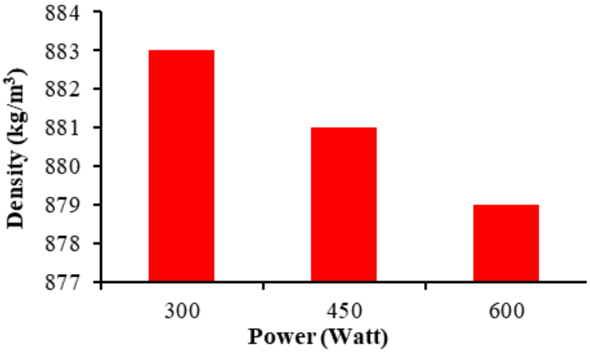

Figure 6. Biodiesel density chart for each power variation

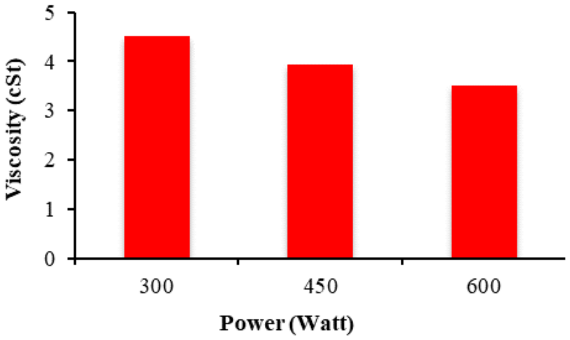

Figure 7. Biodiesel kinematic viscosity chart for each power variation

3.5. Biodiesel Characterization Analysis Using GC-MS

In the 600-watt biodiesel, three methyl ester fatty acid components exhibit dominant peaks, surpassing the intensity of other methyl ester fatty acid components. The three fatty acid methyl ester components with the highest composition percentage or dominant peak are hexadecanoic acid, methyl ester (22.664%) from the MS spectrum having a molecular weight of m/z = 270 and the basic peak in the target compound is m/z = 74, 9,12-Octadecadienoic acid (Z,Z)-methyl ester (30.176%) from the MS spectrum has a molecular weight of m/z = 294 and the basic peak in the target compound is m/z = 67. 9- Octadecadienoic acid (Z)-, methyl ester (38.656%) from the MS spectrum, has a molecular weight of m/z = 296 and a basic peak in the target compound of m/z = 55.

Previous research [29] suggests that the biodiesel chromatogram results obtained are not yet pure, indicating that heterogeneous catalysts may have more potential in biodiesel production than homogeneous catalysts. The methyl ester fatty acid composition of the 600-watt biodiesel (FAME content) is measured at 97.73%, aligning with the SNI standards that mandate a minimum methyl ester content of 96.50%.

The type of FAME that can affect the quality or properties of biodiesel is the FAME component, which is classified as a free fatty acid that makes up oil with high levels or is dominant. The conversion of elevated levels of these free fatty acids into methyl ester fatty acids suggests that the resulting biodiesel is purer, aligning more closely with biodiesel standards for density and viscosity. In this research component, fatty acid methyl ester is the most dominant in biodiesel products, with a power of 600 watts. This research is in accordance with the fatty acid content contained in the ingredients. The basic ingredients of candlenut oil are oleic acid, linoleic acid, and palmitic acid [6].

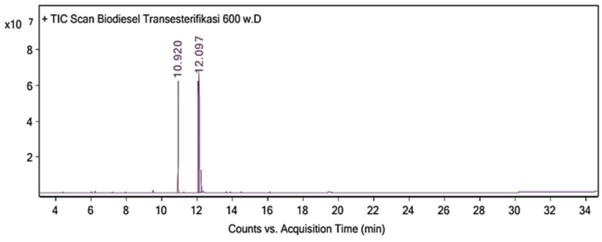

Figure 8. Biodiesel chromatogram result of transesterification power at 600 watts

4. Conclusion

The synthesis of the NaOH/CaO/CA catalyst using the base impregnation method has led to an increased catalyst surface area compared to a single CaO catalyst, as evidenced by a surface area of 9.306 m^2/g obtained from BET analysis. The choice of power during the transesterification process significantly influences the characteristics of the resulting biodiesel. Among the three power levels investigated (300, 450, and 600 watts), the dominant power for producing biodiesel with the highest yield was determined to be 600 watts, achieving an impressive 85.625% yield. Furthermore, this biodiesel exhibited density and viscosity values (879 kg/m^3 and 3.51 cSt, respectively) that are most in line with the standards set by SNI 2341. The GC-MS analysis of the biodiesel produced at 600 watts indicated a methyl ester content of 97.73%, aligning well with the SNI biodiesel standard of 96.50%. These comprehensive results underscore the effectiveness of the NaOH/CaO/CA catalyst and the impact of power settings on optimizing biodiesel production with favorable characteristics and meeting stringent regulatory standards.

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