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MICROWAVE-ASSISTED TRANSESTERIFICATION FOR BIODIESEL PRODUCTION: CATALYST ACTIVITY OF Ca/Na/γ-Al_2O_3 DERIVED FROM EGG SHELLS

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Abstract- Biodiesel is a vegetable fuel produced from the reaction between vegetable oils with mono-alkyl alcohol with catalyst. Coconut oil biodiesel is a mixture rich in medium chain saturated methyl esters and presents similar properties to diesel. Biodiesel production has been carried out using conventional heating. One alternative that is used and is effective in the heating process is microwaves. Wave heating can reduce reaction time and is effective in the manufacturing process. This research aims to encourage the activity of the CaO/Na/γ-Al_2O_3 catalyst to produce biodiesel from coconut oil using microwave. The reaction process was performed with variations in catalyst concentrations of 1; 1.5; 2; 2.5; and 3% at 400 W of power with a reaction time of 7.5 mins. The yield of biodiesel products is compared with biodiesel specifications PT. Pertamina, were determined viscosity, density, flash point, acid number, and cetane number. The biodiesel catalyst concentration of 2.5% with a time of 7.5 minutes from coconut oil and methanol with a catalyst and using microwave heating obtained a biodiesel product yield of 91.84% with density quality specifications of 0.874 g/cm^3, viscosity 2.62 cSt, acid number 0.38 mg KOH/g, cetane number 61, and flash point 135 °C.

Keywords: Biodiesel, Catalyst, Coconut Oil, Microwave, Transesterification.

1. INTRODUCTION

The worldwide shortage of fossil fuels has emerged as a significant global concern for ensuring energy security, as it is projected that oil reserves will be exhausted by the year 2050. As a result, finding sustainable energy resources is critical. Biodiesel stands out as a highly promising substitute for diesel obtained from fossil fuels, thanks to its elevated flash point, excellent biodegradability, and renewable nature. Biodiesel is more environmentally friendly than petroleum diesel. It emits far fewer greenhouse gases and may be utilized in diesel engines without further modification [1]. The rapid increase in energy consumption, especially in the transportation sector, has led to an energy crisis that has resulted in a decrease in fossil fuel supplies and an increase in environmental pollution [2]. Biodiesel is the best alternative to diesel fuel. It is not only compatible with the machine without conversion, but it is also environmentally friendly. Biodiesel can be generated from animal fat or vegetable oil. Coconut oil has excellent properties and supports being processed as a raw material for the production of renewable fuels. One reaction to reduce the viscosity of coconut oil is the transesterification reaction.

The transesterification reaction is a reaction between triglycerides and alcohol to produce free glycerol and fatty acid alkyl esters. The transesterification reaction takes place with the help of a base or acid catalyst. Base catalysts are generally more widely used than acid catalysts [3]. At the same temperature and concentration, metanalysis is faster with a base catalyst than with an acid catalyst [4]. One effort that can be made to overcome this situation is to use an alkaline catalyst [5]. Several studies show that using a solid catalyst produces a higher product yield but requires a large concentration and a long time, so the quality of the methyl ester product and glycerol by-product is better. The use of an acid catalyst requires the longest reaction time and a relatively higher reaction temperature [6]. The role of the catalyst that influences the conversion is the desired methyl ester yield.

In a reaction, the ideal or expected characteristics of a catalyst are active, selective, stable, and economical. Active means it can speed up the reaction to form intermediate products due to interactions between reactants. Selective means it can increase the main product and reduce the side products of the catalysis reaction. The development of the reaction process can be carried out using electromagnetic wave energy [7]. Microwave-assisted transesterification (MAT) has the advantage of more even heating because it does not transfer heat from outside but generates heat from within the material. The second mechanism in microwave heating is ionic conduction, which occurs in compounds containing ions. If a solution containing charged particles or ions is given an electric field, the ions will move.

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This movement will result in an increase in the speed of the collision, thereby converting kinetic energy into heat energy [8]. In a reaction process with polar reagents, if you use a microwave, the heating effect will directly affect the extracted material and also the reagents, usually called volumetric heating [9]. Microwaves are a source of energy that can be used in a reaction, which can accelerate the reaction process [10].

Transesterification with a base catalyst can be 4000 times faster than using the transesterification process with an acid catalyst. However, this process is very sensitive to the quality of the raw materials; it requires raw materials with low FFA and low water content. Conventionally, the manufacture of biodiesel from microalgae consists of two stages, namely extraction and transesterification, which require a large amount of solvent as well as high energy and operational costs. Numerous research studies have been undertaken to eliminate the extraction process by employing techniques such as in situ transesterification or direct transesterification methods [11]. Alternative catalyst options for biodiesel production encompass robust alkali catalysts such as NaOH, KOH, CH_3ONa, and CH_3OK [12].

The in-situ transesterification process is a reaction in which the oil stored in the material comes into direct contact with the alkalized or acidified alcohol, this involves a pre-extraction reaction with the alcohol, where both extraction and transesterification occur simultaneously, with alcohol serving as both a solvent and a reagent. Several previous studies have been carried out using in situ transesterification with the utilization of an acid catalyst [13]. Meanwhile, MAT is a technique for extracting materials dissolved in materials with the help of microwave energy. Microwaves can be used as a heater, which is very effective and does not require the use of a large catalyst. Microwave emit electromagnetic wave energy that penetrates directly into the food container (without heating it, so no energy is wasted heating the food container) and then the food from the inside. The performed research heat transfer from microwaves into the material through the mechanisms of dipolar polarization, ion conduction, and interfacial polarization causes rapid superheating of the reacting material. In an in-situ reaction, the alcohol molecule has a dipole moment, which then tries to have the same electric dipole moment [14].

Meanwhile, the increased product yield and efficiency are achieved through the use of organic acid, which are among the most commonly utilized acids as catalysts in transesterification. Meanwhile, using hydrochloric acid are favored as catalyst in the production of biodiesel, the highest tar efficiency [15]. MAT presents a promising alternative for biodiesel production, significantly reducing the reliance on both homogeneous and heterogeneous catalysts while optimizing reaction time and conditions.

This method not only enhances yield but also improves energy efficiency, challenges such as non-homogeneous electromagnetic fields and limited microwave penetration depth can affect mass transfer efficiency. If the transesterification reaction process can be carried out using a microwave as an energy source, it can reduce the use of homogeneous and heterogeneous catalysts in a short time and atmospheric pressure more efficient.

In Microwave-Assisted Transesterification (MAT) process, $CaO/Na/\gamma\text{-}Al_2O_3$ can serve as a catalyst and have a notable impact on the effectiveness of producing liquid biodiesel products. The advantage of this research is using a $CaO/Na/\gamma\text{-}Al_2O_3$ catalyst, with the main ingredient being duck egg shells, which are processed to produce the catalyst [13, 16].

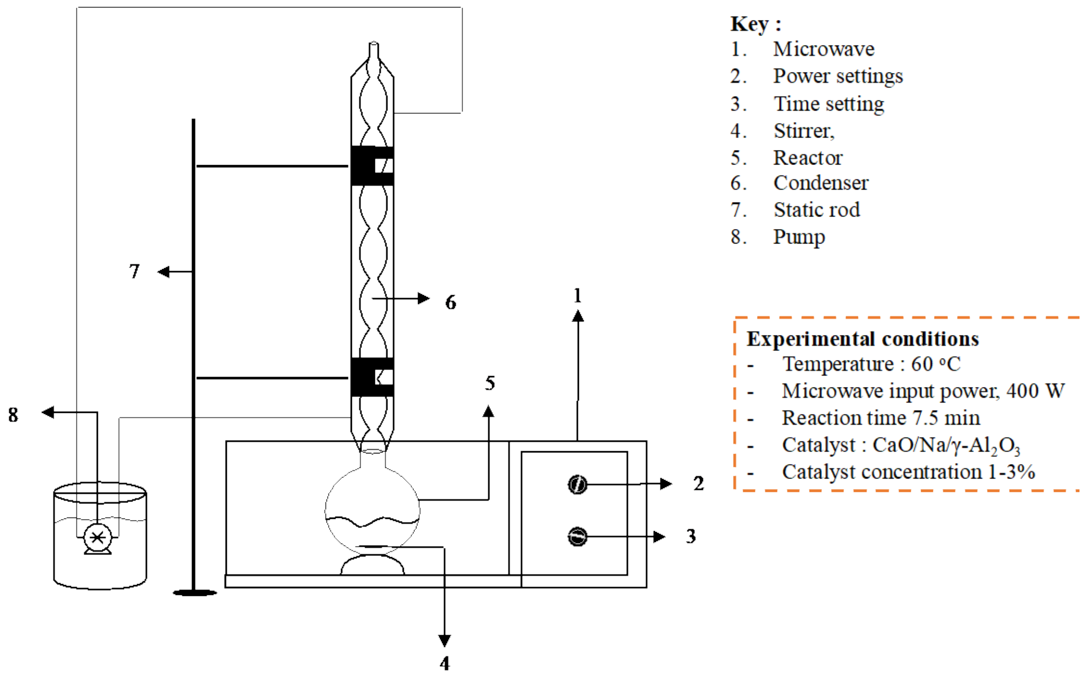

Figure 1. Schematics illustration of the microwave reactor in situ transesterification

2. MATERIAL AND METODS

2.1. CaO Catalyst from Egg Shells Preparation

The materials used include duck egg shells, Barco brand coconut oil, methanol, NaOH, Gamma Alumina, and DI-water. All analytical grade products for all chemicals required without further purification. After being rinsed with water, duck egg shells are sun-dried for 12 hours. Afterward, it was desiccated in a 110 °C oven for ten hours. Subsequently, the powdered duck egg shells are ground into a powder and passed through a 200-mesh sieve. Then the egg-shell powder is calcined in a furnace at 900 °C for 6 hours until solid-set CaO is produced [10, 17].

2.2. Impregnation of Duck Egg Shells with NaOH and γ-Al_2O_3

Duck egg shell powder that has been calcined is impregnated using 1 gram of solid NaOH (pa) and 5 grams of γ-Al_2O_3 then 40 ml of DI-water is added. The mixture was stirred until it was homogeneous and became a paste. Place in the oven at 110 °C for 8 hours. The CaO/Na/γ-Al_2O_3 solid formed was heated at 500 °C for 3 hours. After that, it is ground again and sieved with a 200-mesh sieve.

2.3. Production of Biodiesel

Coconut oil in as much as 50 ml for 30 mins at a temperature of 60 °C. In addition, 24 ml of methanol and 1% CaO/Na/γ-Al_2O_3 catalyst were added to the coconut oil and stirred process for 1 hour. It was then deposited for three hours in a separating funnel, which separated the biodiesel in the bottom layer. Biodiesel is purified through an oven preheating process at 110 °C for 1 hour. This procedure is repeated according to the specified variables, and gas chromatography is used to analyze the biodiesel product for methyl ester composition. The design of the biodiesel manufacturing process equipment is as shown in Figure 1.

3. RESULT AND DISCUSSION

3.1. Preparation of CaO Catalyst from Egg Shells

The process of producing catalysts from duck egg shells is shown in its concrete appearance in Figure 2. The test results show that $CaCO_3$ containing duck egg shells decompose into CaO after a six-hour heating process at 900 °C CaO production utilizes pulverized egg shells as the primary component, as illustrated in Figure 3. Egg shells have the same amount of CaO as the catalyst that is made when γ-Al_2O_3 and NaOH solutions are mixed together. This impregnation is to attach Na and alumina metal components. The addition of NaOH to CaO will increase the basicity of the catalyst because Na^+ ions enter the catalyst cavities, which can increase the basicity of the catalyst. Meanwhile, the addition of γ-Al_2O_3 functions as a catalyst support, which can improve the performance of a catalyst [10, 17]. Then add distilled water to make the stirring process easier so that the mixing process is homogeneous. Next, heating is carried out using an oven at 110 °C for 8 hours so that the water content in the catalyst can be evaporated, followed by a calcination process at that temperature. Next, calcination is carried out, and it is hoped that this can evaporate unwanted compounds. The eggshell-based CaO/Na/γ-Al_2O_3 catalyst product, shown in Figure 2, is ready to be used to rate up the reaction. The assessment of catalyst activity was conducted on the transesterification process involving methanol and coconut oil for the production of biodiesel.

Figure 2. Egg shells, the basic material for producing catalysts:
a. egg shell; b. CaO/Na/γ-Al_2O_3

3.2. X-Ray Fluorescence Analysis

The CaO/Na/γ-Al_2O_3 catalyst product obtained was tested using X-ray fluorescence analysis for the elemental content of the catalyst. The analysis results can be showed in Table 1.

Table 1. The analysis of CaO/Na/γ-Al_2O_3 catalyst using XRF

Parameter	Rate (%)
SiO_2	0.07
Al_2O_3	28.65
TiO_2	0.02
Fe_2O_3	< 0.001
CaO	52.83
MgO	0.37
K_2O	0.04
Na_2O	1.52
Na	1.12

The analysis results show that the elements Na and CaO are well distributed into the γ-Al_2O_3 compound after calcination at a temperature of 900 °C. This is proven by the XRF analysis shown in Table 1. The catalyst composition contains 52.83% CaO, 28.65% γ-Al_2O_3, and 1.12% sodium. With high CaO content, duck egg shells have the potential to be an economical source of CaO and can be used as a source of CaO catalyst [18].

3.3. Scanning Electron Microscopy (SEM) Analysis

The completed catalyst was evaluated using SEM and EDS analysis. The SEM-EDS test was carried out to determine the surface morphology and chemical composition of the CaO/Na/γ-Al_2O_3 catalyst in Figure 3 depicts the shape of the catalyst developed in this research.

The evaluate of the SEM analysis illustrate that the appearance of the catalyst has a clean surface appearance and shows a high degree of crystallinity.

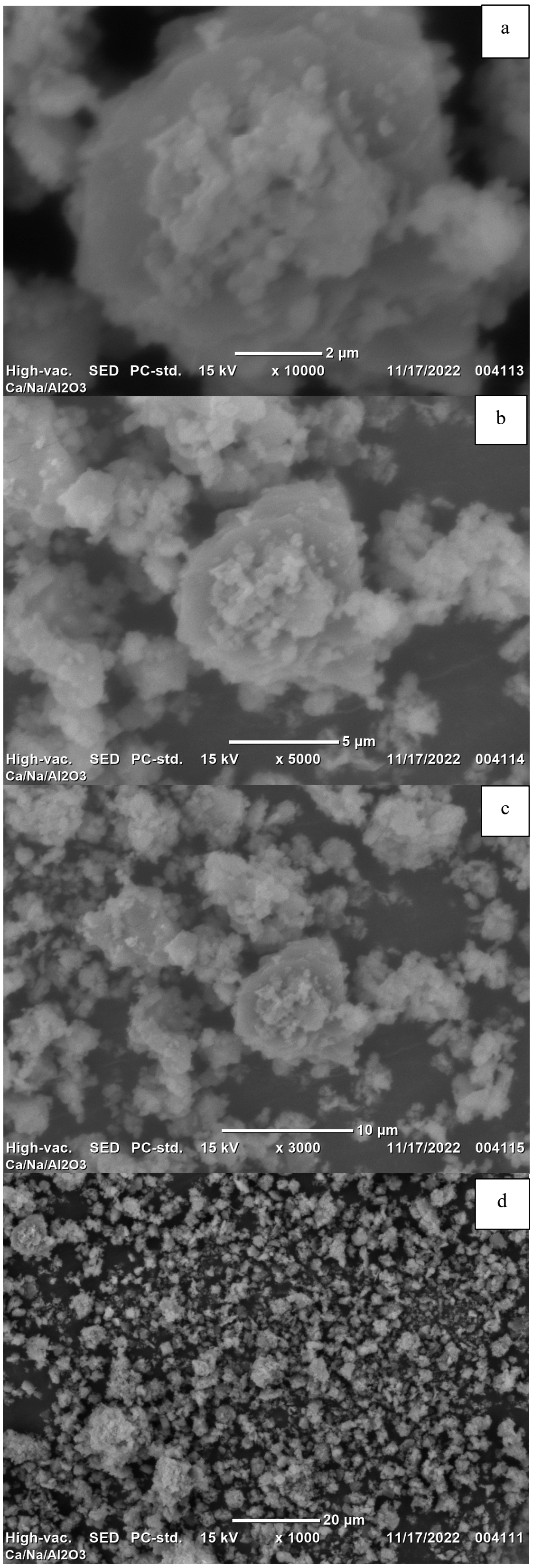

Figure 3. Catalyst Surface Morphology, a) 2000*x* magnification, b) 5000*x* magnification, c) 10000*x* magnification, d) 20000*x* magnification

The activity of catalysts is significantly influenced by their structural characteristics. Understanding these relationships is crucial for optimizing catalyst performance [19]. The crystal structure of the CaO/Na/γ-Al_2O_3 catalyst looks irregular (irregular shape). The CaO pores are no longer visible because they are filled with NaOH, indicating that Na was thoroughly decomposed during the calcination process. Figure 3 shows that the CaO/Na/γ-Al_2O_3 catalyst contains 74.05% CaO compounds, of which the Ca element is 58.92 and the γ-Al_2O_3 compound is 5.22%, of which the Al element is 4.57%, and the total mass of the oxygen element is 33.81%.

3.4. CaO/Na/γ-Al_2O_3 Catalyst Activation Test with Biodiesel Production

The CaO/Na/γ-Al_2O_3 catalyst performance was tested in a transesterification reaction with coconut oil and methanol in order to make biodiesel. The catalyst was utilized at concentrations of 1%, 1.5%, 2.5%, and 3% (w/v) and was heated in a microwave at 400 W with a time variation of 2.5, 5, 7.5, 10, and 15 minutes. The product that remains is subsequently filtered through a separating funnel. The biodiesel formed is then washed using distilled water, and the resulting biodiesel product is placed in the oven at 110 °C for one hour.

3.5. Catalyst Concentration of CaO/Na/γ-Al_2O_3 on Biodiesel Yield

The results of the catalyst activity test were conducted at different concentrations of 3%, 1.5%, 2%, 2.5%, and 1%. The resulting biodiesel products are displayed in Figure 4.

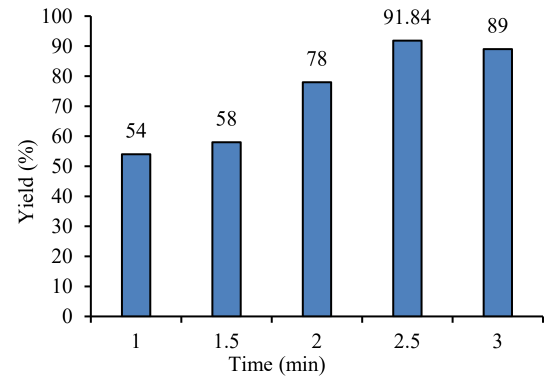

Figure 4. Effect of catalyst concentration on the production of methyl ester products

From Figure 4 shows the results of the addition 1% catalyst the resulting biodiesel yield was 54%, 1.5% catalyst was 58%, 2% catalyst was 68%, 2.5% catalyst was 78% and 3% catalyst with 72%. This demonstrates that increasing of catalysts biodiesel products. Increasing the number of catalysts greatly influences the yield because the catalyst will expand the contact area between reactants so that the activation energy will increase, causing the reaction rate to increase [20]. It can be seen that from various variations in catalyst concentration, the smallest yield was obtained at a catalyst concentration of 1% and the largest yield was obtained at a catalyst concentration of 2.5%.

3.6. Effect of Reaction Time on Yield in Biodiesel Products

The production of biodiesel, catalysts are chemically important in the breakdown of molecular bonds to form new ones, in this case, methyl. The impact of reaction or response time on the produced biodiesel yield is illustrated in Figure 5, in accordance with the findings of the conducted research.

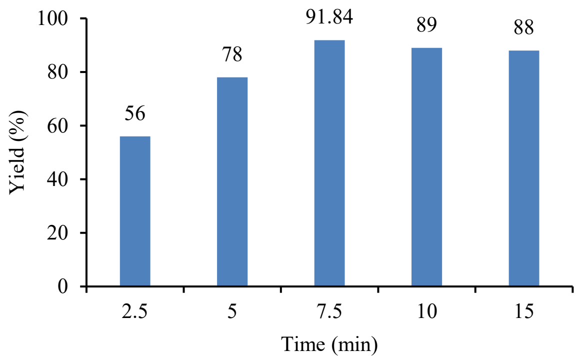

Figure 5. The relationship between the effect of reaction time on the yield of biodiesel products

From Figure 5 the increase in biodiesel yield from 2.5 to 5 and peaked at 7.5 minutes, while at 10 minutes and 15 it tended to decrease. This shows that the transesterification reaction of coconut and methanol was completed in 7.5 minutes. This can be said by increasing the reaction time to convert coconut oil and methanol into biodiesel through transesterification reactions, causing an increase in the yield of the biodiesel product produced [21]. The power input to the microwave affects the wave amplitude. The rotation speed of polar molecules has a linear relationship to the microwave amplitude [22]. The greater the biodiesel yield will be.

Meanwhile, at 10 minutes, the biodiesel yield decreased. This is due to the temperature factor in the reaction. High microwave power will cause an excessive increase in the reaction temperature and evaporation of methanol. This will cause methanol to evaporate, thereby reducing the methanol content in the reactants [8, 23]. This decrease will cause a reduction in the reaction between triglycerides and methanol, so the amount of biodiesel produced will decrease. Methanol is essential for the transesterification of triglycerides into fatty acid methyl esters (FAME) [24]. A higher methanol concentration typically shifts the reaction equilibrium towards biodiesel production. When methanol is reduced, particularly below a critical threshold (6-8 wt. %), a double-liquid phase can form, which hinders effective mixing and reduces biodiesel yield.

In contrast, while reducing methanol may seem detrimental, some studies suggest that optimizing methanol use can lead to more efficient processes, particularly when employing advanced catalytic methods [11, 25].

3.7. Effect of CaO/Na/γ-Al_2O_3 Catalyst Concentration on Viscosity in Biodiesel Products

One of the parameters that physically indicates the formation of biodiesel products from the transesterification reaction between triglycerides and methanol is a decrease in viscosity. The results of measuring the viscosity of biodiesel are shown in Figure 6.

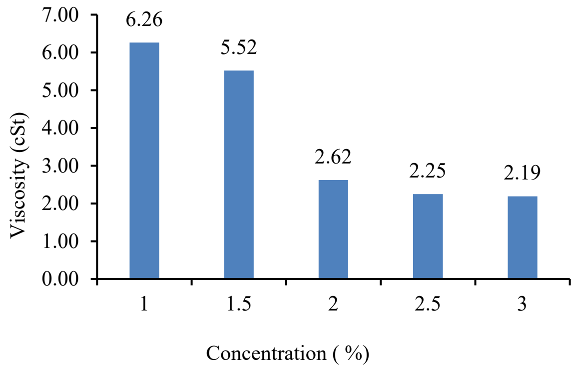

Figure 6. The Effect of catalyst concentration on the viscosity of biodiesel products at 400 W

According to Figure 6, the utilization of the CaO/Na/γ-Al_2O_3 catalyst at a concentration of 1% to 2% results in a significant decrease. At a concentration of 1%, the viscosity is 6.26 cSt. At concentrations of 2 to 3%, the viscosity remains rather constant, namely at 2.25 and 2.19 cSt. The optimal sample concentration of 2.5% yielded a viscosity of 2.25 cSt.

3.9. Characterization of Biodiesel Products from Coconut Oil

Biodiesel products are analyzed using a Gas Chromatography instrument to determine the components of the product compounds in Figure 7. From Figure 7, is a chromatogram from the results of gas chromatography analysis of biodiesel products obtained with a catalyst concentration of 2%, a reaction time of 7.5 minutes with power 400. The analysis results show that the chromatogram shows that methyl ester products are formed from various fatty acids. There are six dominant hydrocarbon methyl esters, including: methyl capric, methyl lauric, methyl myristic, methyl palmitic, methyl oleic, and methyl stearic [26].

Overall, the methyl ester product obtained from the research results shows that it meets the standards of the specified parameters. The following factors are used to determine the quality criteria for biodiesel products: density, viscosity, acid number, cetane number, and flash point [27, 28]. Methyl esters, particularly those derived from palm oil, exhibit similar gas compositions to mineral oils during electrical stress but at higher concentrations. This necessitates adjustments in existing fault interpretation methods for effective monitoring [29]. Qualitative analysis using gas chromatography and mass spectrometry has proven effective in distinguishing between various methyl esters based on their fragmentation patterns and retention indices, enhancing the accuracy of identification [30].

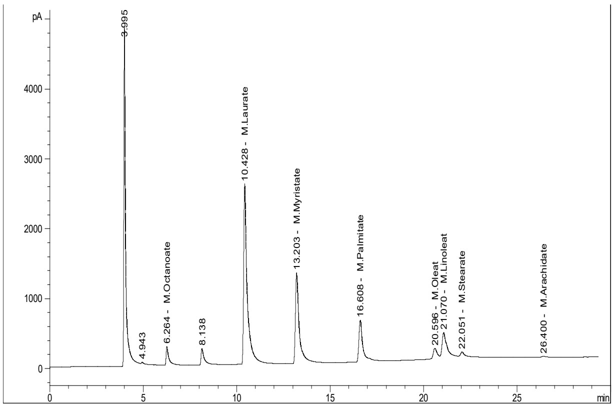

Figure 7. Chromatogram of Biodiesel product analysis Gas Chromatography

Table 2. Analyses of Methyl ester products in comparison to standard methyl esters

Parameters	Product	SNI-7182
Density (40 ºC), g/ml	0.874	0.85-0.89
Kinematic Viscosity (40 ºC), cSt	2.62	2-6
Acid Number, mg-KOH/g	0.38	max 0.4
Cetane Number, --	61	min 51
Flash Point, ºC	135	min 130

4. CONCLUSIONS

In this research, the activity of the CaO/Na/γ-Al_2O_3 as catalyst increases in the manufacture of biodiesel was done with Microwave-assisted transesterification (MAT). The reaction process, variations of catalyst concentrations input power, reaction time and the yield of biodiesel was evaluated. In comparison to conventional or non-microwave catalytic pyrolysis, the MAT processes can reduce the amount of catalyst and higher the reaction process. The highest biodiesel product yield was achieved with a catalyst concentration of 2.5%, a reaction time of 7.5 minutes, and 400 W of microwave power, the resulting biodiesel product is 91.84 percent in volume, with density quality specifications of 0.874 g/cm^3 and conforms to SNI-7182 product standard. Thus, this study becomes basis for reducing global carbon emissions by green energy.

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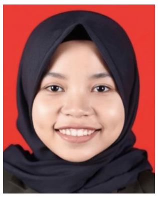

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