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Catalyst activity of Ca/Na/ γ -Al₂O₃ for Biodiesel Production using Microwave

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Abstract- Biodiesel is a vegetable fuel produced from the reaction between vegetable oil and methanol with the help of a catalyst. Several studies have proven that coconut oil can be used as a raw material for making biodiesel. 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 determine the activity of the CaO/Na/ γ -Al₂O₃ catalyst in making biodiesel from coconut oil using microwave. The reaction process was carried out with variations in catalyst concentrations of 1; 1.5; 2; 2.5; and 3% at 400 watts of power with a reaction time of 7.5 minutes. The yield of biodiesel products is compared with biodiesel specifications from Pertamina, including density, viscosity, acid number, flash point, and cetane number. The research results showed that the best biodiesel product at a 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 8.74 gr/cm³, viscosity 2.62 cSt, acid number 0.38 mg KOH/gr, cetane number 61, and flash point 135 °C.

Keywords: Coconut Oil, biodiesel, catalyst, microwave, transesterification.

1. INTRODUCTION

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 produced from vegetable oil or animal fat. 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 [1].

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 [2]. At the same temperature and concentration, methanolysis is faster with a base catalyst than with an acid catalyst [3]. One effort that can be made to overcome this situation is to use an alkaline catalyst [4]. 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 [5]. 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 [1]. Microwave heating 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. This movement will result in an increase in the speed of the collision, thereby converting kinetic energy into heat energy [7]. The advantages of using microwaves can be explained by the fact that microwave radiation has been proven to be a very effective heating source in chemical reactions. Microwaves can speed up the reaction speed, resulting in better product yields because microwave heating applies directly to the material [1]. 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 [8]. Microwaves are a source of energy that can be used in a

reaction, which can speed up the reaction process [9]. 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, making it better and more efficient. The advantage of this research is using a CaO/Na/ γ -Al₂O₃ catalyst, with the main ingredient being duck egg shells, which are processed to produce the catalyst. We request authors to follow this guideline and format their manuscripts exactly the same as this document.

2. MATERIAL AND METODS

2.1 Materials

The materials used include duck egg shells, Barco brand coconut oil, methanol, NaOH, Gamma Alumina, and distilled water.

2.2 Methods

2.2.1 Preparation of CaO Catalyst from Egg Shells

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 a temperature of 900 °C for 6 hours until solid CaO is produced.

2.2.2 Impregnation of Egg Shells with NaOH and γ -Al₂O₃

Duck egg shell powder that has been calcined is impregnated using 1 gram of solid NaOH (pa) and 5 grams of γ -Al₂O₃ then 40 ml of distilled water is added. The mixture was stirred using a magnetic stirrer until it was homogeneous and became a paste. Place in the oven at 110 °C for 8 hours. The CaO/Na/ γ -Al₂O₃ solid formed was heated at 500 °C for 3 hours. After that, it is ground again and sieved with a 200-mesh sieve.

2.2.3 Production of Biodiesel

Coconut oil in as much as 50 ml for 30 minutes at a temperature of 60 °C. Then 24 ml of methanol and 1% CaO/Na/ γ -Al₂O₃ catalyst were added to the coconut oil and stirred using a magnetic stirrer 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.

2.2.4 Equipment Design

The design of the biodiesel manufacturing process equipment is as shown in (Fig.1) All material on all pages should fit within an area of A4 (21 x 29.7 cm), 2.8 cm from the top of the page and ending with 2.4 cm from the bottom.

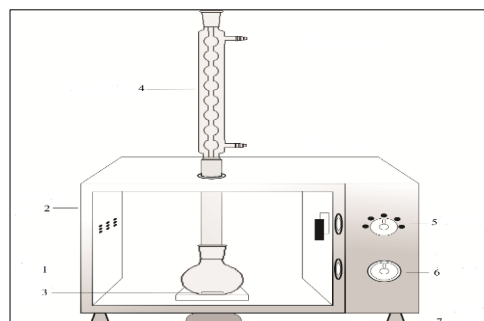


Fig.1 Schematic illustration of microwave reactor in situ transesterification; 1. Reactor, 2. Microwave, 3. Bar stir, 4. Condenser, 5. Power Setting, 6. Time setting, 7. Magnetic stirrer.

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 3.1. The test results show that CaCO₃ containing duck egg shells decompose into CaO after a six-hour heating process at 900 degrees Celsius. CaO production utilizes pulverized egg shells as the primary component, as illustrated in (Fig. 3.1.a) Egg shells have the same amount of CaO as the catalyst that is made when γ -Al₂O₃ and NaOH (pa) 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 -Al₂O₃ functions as a catalyst support, which can improve the performance of a catalyst [10]. 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 a temperature of 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 -Al₂O₃ catalyst product, shown in (Fig.3.1.b), is ready to be used to speed up the reaction. The assessment of catalyst activity was conducted on the transesterification process involving methanol and coconut oil for the production of biodiesel.



Fig. 2 Egg shells, the basic material for producing catalysts: a. egg shell; b. $\text{CaO}/\text{Na}/\gamma\text{-Al}_2\text{O}_3$

3.2. X-Ray Fluorescence AnalysisTypeset Text

The $\text{CaO}/\text{Na}/\gamma\text{-Al}_2\text{O}_3$ catalyst product obtained was tested using X-ray fluorescence analysis for the elemental content of the catalyst. The analysis results can be seen in Table 1.

Table 1. The analysis of $\text{CaO}/\text{Na}/\gamma\text{-Al}_2\text{O}_3$ 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 $\gamma\text{-Al}_2\text{O}_3$ compound after calcination at a temperature of 900 °C. This is proven by the results of the XRF analysis shown in Table 1. The catalyst composition contains 52.83% CaO, 28.65% $\gamma\text{-Al}_2\text{O}_3$, 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 [11].

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 $\text{CaO}/\text{Na}/\gamma\text{-Al}_2\text{O}_3$ catalyst (Fig. 3) depicts the shape of the catalyst developed in this research.

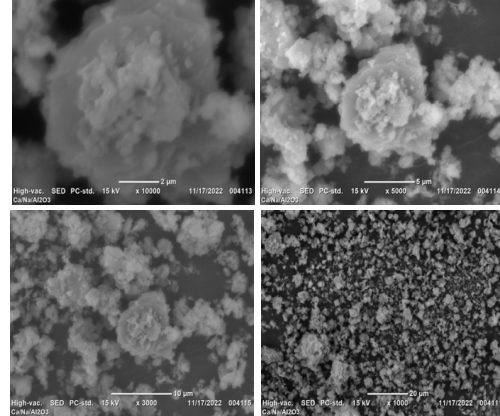


Fig. 3 Catalyst Surface Morphology (a) 2000x magnification, (b) 5000x magnification, (c) 10000x magnification, (d) 20000x magnification

The results of the SEM analysis illustrate that the appearance of the catalyst has a clean surface appearance and shows a high degree of crystallinity. The crystal structure of the $\text{CaO}/\text{Na}/\gamma\text{-Al}_2\text{O}_3$ 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 3.3 shows that the $\text{CaO}/\text{Na}/\gamma\text{-Al}_2\text{O}_3$ catalyst contains 74.05% CaO compounds, of which the Ca element is 58.92 and the $\gamma\text{-Al}_2\text{O}_3$ 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. $\text{CaO}/\text{Na}/\gamma\text{-Al}_2\text{O}_3$ Catalyst Activation Test with Biodiesel Production

The $\text{CaO}/\text{Na}/\gamma\text{-Al}_2\text{O}_3$ 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 watts 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 a temperature of 110 °C for one hour.

3.5 Effect of $\text{CaO}/\text{Na}/\gamma\text{-Al}_2\text{O}_3$ Catalyst Concentration on Biodiesel Yield

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

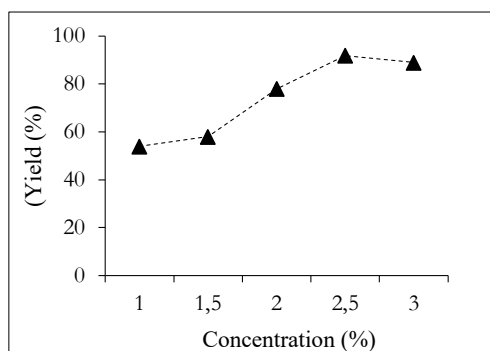


Fig. 4 Effect of catalyst concentration on the production of methyl ester products

(Fig. 4) shows that by adding 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 was 72%. This demonstrates that increasing the number of catalysts increases the number of 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 speed to increase [12]. 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

In 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 time on the produced biodiesel yield is illustrated in (Fig. 5), in accordance with the findings of the conducted research.

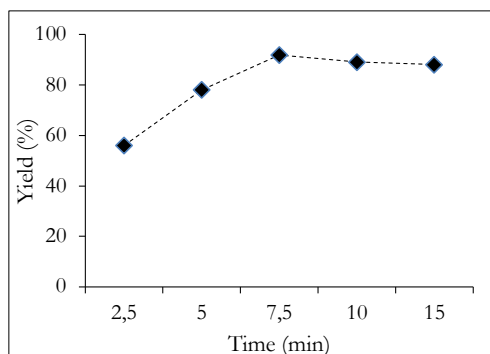


Fig.5 The relationship between the effect of reaction time on the yield of biodiesel products

(Fig. 5) showed that there was an 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 [13]. The power input to the microwave affects the wave amplitude. The greater the power used to generate microwaves, the greater the electric field produced [14]. The rotation speed of polar molecules has a linear relationship to the microwave amplitude [15]. The longer the reaction time and the higher the temperature given, 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. This decrease will cause a reduction in the reaction between triglycerides and methanol, so the amount of biodiesel produced will decrease. The temperature has passed the boiling point of methanol, so some of the methanol undergoes a phase change from liquid to gas. This change in the methanol phase causes the amount of methanol in the liquid phase to decrease. Reducing the amount of methanol in the solution causes a reduction in the number of effective collisions to produce biodiesel, so the yield of biodiesel formed will decrease [1, 16]

3.7. Effect of CaO/Na/γ-Al₂O₃ 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 products are displayed in (Fig. 6) below :

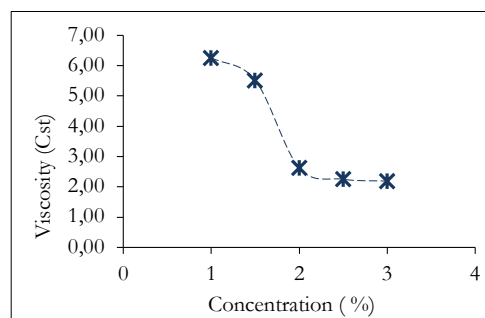


Fig. 6 Effect of catalyst concentration on the viscosity of biodiesel products at 400 watts

According to (Fig. 6) the utilization of the CaO/Na/ γ-Al₂O₃ 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. The results of the analysis can be seen in (Fig. 7).

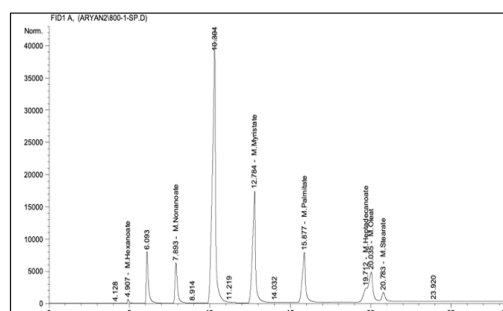


Fig.7 Chromatogram of Biodiesel product analysis Gas Chromatography

(Fig.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, and a microwave power of 400 watts. 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. 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 [17].

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

Parameter	Product	SNI-7182
Density (40 °C) , g/ml	0.874	0.85-0.89
kinematic viscosity (40°C), cSt	2.62	2-6
Acid Number mg-KOH/gr	0.38	Max 0.4
Cetane Number, --	61	Min 51
Flash point, °C	135	Min 130

4. CONCLUSIONS

The activity of the $\text{CaO}/\text{Na}/\gamma\text{-Al}_2\text{O}_3$ catalyst increases in the manufacture of biodiesel with the help of microwave heating. Microwaves can reduce the amount of catalyst and speed up 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 watts of microwave power; the resulting biodiesel product is 91.84 percent in volume and conforms to the SNI-7182 standard.

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Faculty of Industrial Technology, Universitas Muslim Indonesia Makassar.

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Catalyst activity of $\text{Ca}/\text{Na}/\gamma\text{-Al}_2\text{O}_3$ for Biodiesel Production using Microwave

Abstract- Biodiesel is a vegetable fuel produced from the reaction between vegetable oils with mono-alkyl alcohol with catalyst. Several studies have proven that coconut oil can be used as a raw material for making biodiesel. 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 $\text{CaO}/\text{Na}/\gamma\text{-Al}_2\text{O}_3$ catalyst in making 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 watts of power with a reaction time of 7.5 mins. The yield of biodiesel products is compared with biodiesel specifications PT. Pertamina, including viscosity, density, flash point, acid number, and cetane number. The results revealed that the best biodiesel product at a 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 8.74 gr/cm³, viscosity 2.62 cSt, acid number 0.38 mg KOH/gr, cetane number 61, and flash point 135 °C.

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

1. INTRODUCTION

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 [1].

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 [2]. At the same temperature and concentration, methanolysis is faster with a base catalyst than with an acid catalyst [3]. One effort that can be made to overcome this situation is to use an alkaline catalyst [4]. 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 [5]. 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 [1]. 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. This movement will result in an increase in the speed of the collision, thereby converting kinetic energy into heat energy [7]. 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 [8]. Microwaves are a source of energy that can be used in a reaction, which can rate up the reaction process [9]. 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, making

it better and more efficient. The advantage of this research is using a $\text{CaO}/\text{Na}/\gamma\text{-Al}_2\text{O}_3$ catalyst, with the main ingredient being duck egg shells, which are processed to produce the catalyst.

2. MATERIAL AND METODS

2.1 Materials

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.

2.2 Methods

2.2.1 CaO Catalyst from Egg Shells Preparation

After being rinsed with water, duck egg shells are sundried 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.

2.2.2 Impregnation of Egg Shells with NaOH and $\gamma\text{-Al}_2\text{O}_3$

Duck egg shell powder that has been calcined is impregnated using 1 gram of solid NaOH (pa) and 5 grams of $\gamma\text{-Al}_2\text{O}_3$ 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 $\text{CaO}/\text{Na}/\gamma\text{-Al}_2\text{O}_3$ solid formed was heated at 500°C for 3 hours. After that, it is ground again and sieved with a 200-mesh sieve.

2.2.3 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% $\text{CaO}/\text{Na}/\gamma\text{-Al}_2\text{O}_3$ 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.

2.2.4 Equipment Design

The design of the biodiesel manufacturing process equipment is as shown in (Fig.1).

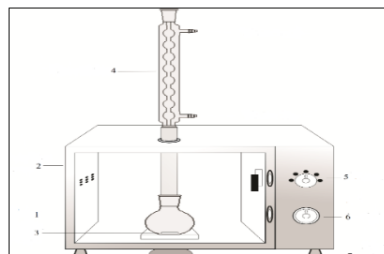


Fig.1 Schematic illustration of microwave reactor in situ transesterification; 1. Reactor, 2. Microwave, 3. Bar stir, 4. Condenser, 5. Power Station, 6. Time Station, 7. Magnetic stirrer.

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 3.1. 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 (Fig. 3.1.a) Egg shells have the same amount of CaO as the catalyst that is made when $\gamma\text{-Al}_2\text{O}_3$ and NaOH (pa) 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 $\gamma\text{-Al}_2\text{O}_3$ functions as a catalyst support, which can improve the performance of a catalyst [10]. 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 $\text{CaO}/\text{Na}/\gamma\text{-Al}_2\text{O}_3$ catalyst product, shown in (Fig.3.1.b), 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.



Fig. 2 Egg shells, the basic material for producing catalysts: a. egg shell; b. $\text{CaO}/\text{Na}/\gamma\text{-Al}_2\text{O}_3$

3.2. X-Ray Fluorescence Analysis

The $\text{CaO}/\text{Na}/\gamma\text{-Al}_2\text{O}_3$ catalyst product obtained was tested using X-ray fluorescence analysis for the elemental

content of the catalyst. The analysis results can showed in Table 1.

Table 1. The analysis of CaO/Na/ γ -Al₂O₃ catalyst using XRF

Parameter	Rate (%)
SiO ₂	0.07
Al ₂ O ₃	28.65
TiO ₂	0.02
Fe ₂ O ₃	< 0.001
CaO	52.83
MgO	0.37
K ₂ O	0.04
Na ₂ O	1.52
Na	1.12

The analysis results show that the elements Na and CaO are well distributed into the γ -Al₂O₃ 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 -Al₂O₃, 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 [11].

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 -Al₂O₃ catalyst (Fig. 3) depicts the shape of the catalyst developed in this research.

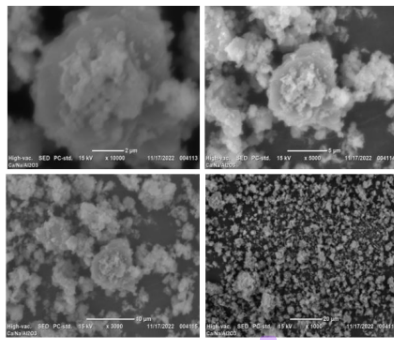


Fig. 3 Catalyst Surface Morphology (a) 2000x magnification, (b) 5000x magnification, (c) 10000x magnification, (d) 20000x magnification

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. The crystal structure of the CaO/Na/ γ -Al₂O₃ 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 3.3 shows that the CaO/Na/ γ -Al₂O₃ catalyst contains 74.05% CaO compounds, of which the Ca

element is 58.92 and the γ -Al₂O₃ 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 -Al₂O₃ Catalyst Activation Test with Biodiesel Production

The CaO/Na/ γ -Al₂O₃ 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 watts 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 -Al₂O₃ on Biodiesel Yield

The 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 (Fig. 4).

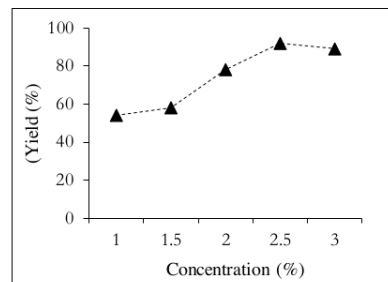


Fig. 4 Effect of catalyst concentration on the production of methyl ester products

(Fig. 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 [12]. 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 (Fig. 5), in accordance with the findings of the conducted research.

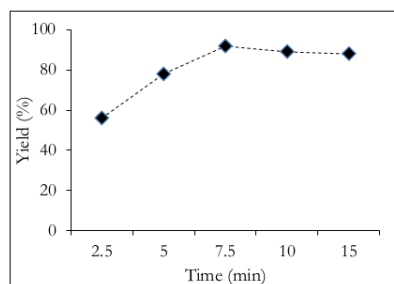


Fig.5 The relationship between the effect of reaction time on the yield of biodiesel products

(Fig. 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 [13]. The power input to the microwave affects the wave amplitude. The rotation speed of polar molecules has a linear relationship to the microwave amplitude [14]. 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 [15]. 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) [16]. 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 [17, 18]

3.7. Effect of CaO/Na/ γ -Al₂O₃ 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 is exhibited in (Fig. 6).

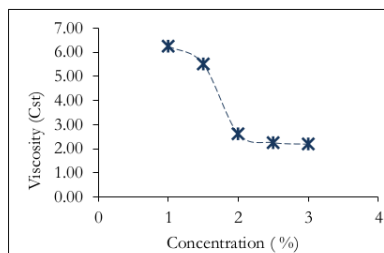


Fig. 6 The Effect of catalyst concentration on the viscosity of biodiesel products at 400 watts

According to (Fig. 6) the utilization of the CaO/Na/ γ -Al₂O₃ 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 (Fig. 7).

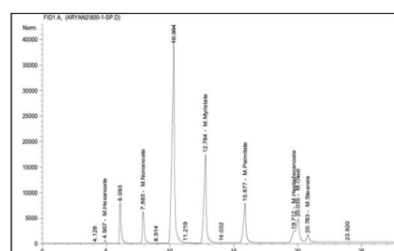


Fig.7 Chromatogram of Biodiesel product analysis Gas Chromatography

(Fig.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 watts. 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. [19]. 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 [20-21].

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

Parameter	Product	SNI-7182
Density (40 °C) , g/ml	0.874	0.85-0.89
kinematic viscosity (40°C), cSt	2.62	2-6
Acid Number mg-KOH/gr	0.38	Max 0.4
Cetane Number, --	61	Min 51
Flash point, °C	135	Min 130

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 [22]. 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 [23].

4. CONCLUSIONS

The activity of the $\text{CaO}/\text{Na}/\gamma\text{-Al}_2\text{O}_3$ catalyst increases in the manufacture of biodiesel with the help of microwave heating. Microwaves can reduce the amount of catalyst and rate up 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 watts of microwave power; the resulting biodiesel product is 91.84 percent in volume and conforms to the SNI-7182 standard.

ACKNOWLEDGEMENTS

The authors gratefully acknowledge the facilities support from the Faculty of Industrial Technology, Universitas Muslim Indonesia Makassar.

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 - **Suggested Title:** "Microwave-Assisted Transesterification for Biodiesel Production: Catalyst Activity of Ca/Na/ γ -Al₂O₃ Derived from Egg Shells."
- **Abstract:** The abstract provides an overview of the study but lacks specific information about the novelty and key findings. The description of the results, particularly the percentages and characteristics of biodiesel yield, needs to be expanded.
 - **Suggested Improvement:** Include the specific advantages of using microwave heating in the abstract and highlight the key performance metrics (e.g., catalyst concentration, reaction time, and yield).

2. Introduction:

- **Content:** The introduction gives a good background on biodiesel production and catalysts but lacks a clear explanation of the **microwave heating** process and its advantages over conventional heating. It should also explain more thoroughly the reason for using **CaO/Na/ γ -Al₂O₃** and how it differs from other catalysts.
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3. Methodology:

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 - Suggested Improvement:** Provide more detailed specifications for equipment used, especially the **microwave reactor**. The conditions for calcination and catalyst preparation need clearer steps, including temperatures, times, and materials used.

To : Editors and Reviewers IJTPE

We would like to thank the editors and reviewers who have given the opportunity to revise this journal. This journal has been revised and has been classified according to reviewer's comments. Several wrong textual errors has been revised also.

Special comments from reviewer :

Coments from Reviewers	Before Revision	Aftre Revision
Title	Title : Catalyst activity of Ca/Na/γ-Al2O3 for Biodiesel Production using Microwave	Title : Microwave-Assisted Transesterification For Biodiesel Production: Catalyst Activity Of Ca/Na/Γ-Al2o3 Derived From Egg Shells
Abstract: The abstract provides an overview of the study but lacks specific information about the novelty and	Abstract- Biodiesel is a vegetable fuel produced from the reaction between vegetable oil and methanol with the help of a catalyst. Several studies have proven that coconut oil can be used as a raw material for making biodiesel. Biodiesel production has been carried	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

key findings. The description of the results, particularly the percentages and characteristics of biodiesel yield, needs to be expanded. Suggested Improvement: Include the specific advantages of using microwave heating in the abstract and highlight the key performance metrics (e.g., catalyst concentration, reaction time, and yield).	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 determine the activity of the CaO/Na/γ-Al₂O₃ catalyst in making biodiesel from coconut oil using microwave. The reaction process was carried out with variations in catalyst concentrations of 1; 1.5; 2; 2.5; and 3% at 400 watts of power with a reaction time of 7.5 minutes. The yield of biodiesel products is compared with biodiesel specifications from Pertamina, including density, viscosity, acid number, flash point, and cetane number. The research results showed that the best biodiesel product at a 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 8.74 gr/cm³, viscosity 2.62 cSt, acid number 0.38 mg KOH/gr, cetane number 61, and flash point 135 oC.	production has been carried out using conventional heating. One alternative that is used and is effective in the heating process is microwaves. Vibration heating can reduce reaction time and is effective in the manufacturing process. This research aims to encourage the activity of the CaO/Na/γ-Al₂O₃ 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 8.74 gr/cm³, viscosity 2.62 cSt, acid number 0.38 mg KOH/gr, cetane number 61, and flash point 135 oC.
Introduction: Content: The introduction gives a good background on biodiesel production and catalysts but lacks a clear explanation of the microwave heating process and its advantages over conventional heating. It should also explain more thoroughly the reason for using CaO/Na/γ-Al₂O₃ and how it differs	Introduction: 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 produced from vegetable oil or animal fat. 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 [1]. 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	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

from other catalysts. Suggested Improvement: Add a short section on why microwaves are preferred in this context and a brief literature review that compares this approach to other methods used in biodiesel production. Clarity: Some sentences could be made more concise. For instance, the discussion on base and acid catalysts could be streamlined. Suggested Improvement: Rephrase long sentences and avoid repetition in explaining why base catalysts are more commonly used than acid catalysts.	catalyst. Base catalysts are generally more widely used than acid catalysts [2]. At the same temperature and concentration, methanolysis is faster with a base catalyst than with an acid catalyst [3]. One effort that can be made to overcome this situation is to use an alkaline catalyst [4]. 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 [5]. 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 [1]. Microwave heating 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. This movement will result in an increase in the speed of the collision, thereby converting kinetic energy into heat energy [7]. The advantages of using microwaves can be explained by the fact that microwave radiation has been proven to be a very effective heating source	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
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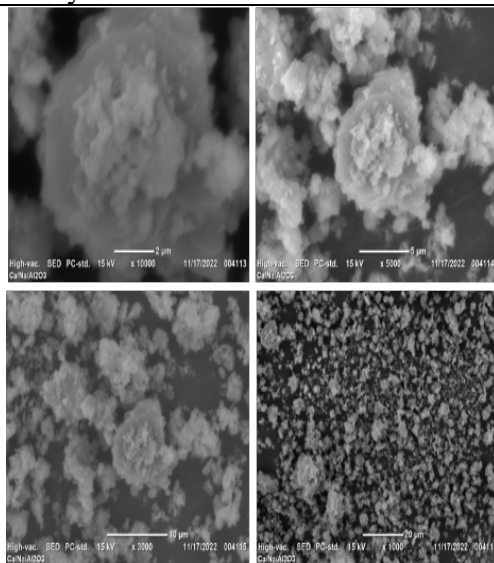
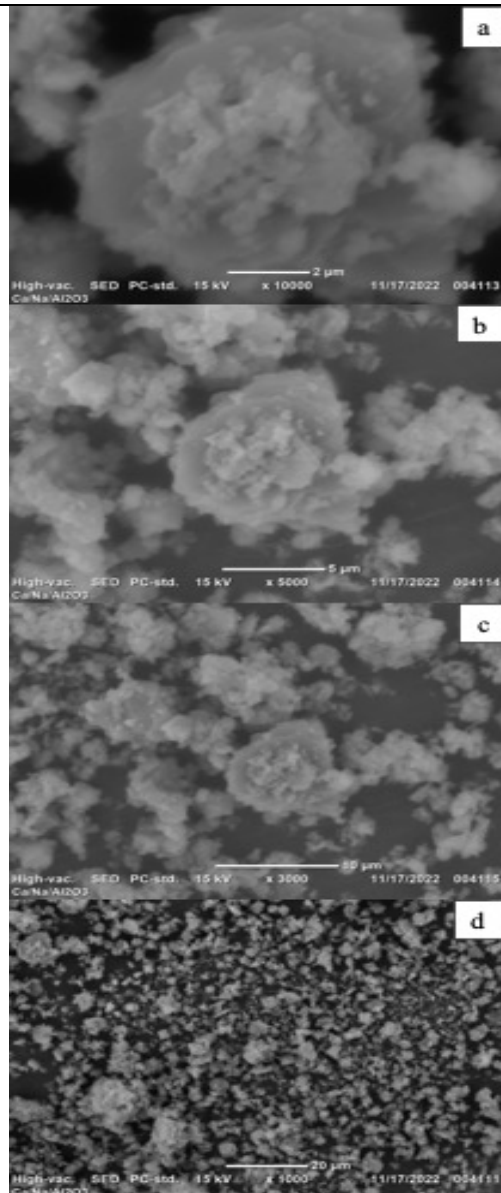
	in chemical reactions. Microwaves can speed up the reaction speed, resulting in better product yields because microwave heating applies directly to the material [1]. In a reaction process with polar reagents, if you use a microwave, the heating effect will directly affect the extracted material and also the reagents, usually called volumetric heating [8]. Microwaves are a source of energy that can be used in a reaction, which can speed up the reaction process [9]. 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, making it better and more efficient. The advantage of this research is using a CaO/Na/ γ-Al₂O₃ catalyst, with the main ingredient being duck egg shells, which are processed to produce the catalyst.	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. Registered: 240604 Article Info: 1901 Accepted: 241114 Available: 241231 Reserved: 240906 70 International Journal on “Technical and Physical Problems of Engineering” (IJTPE), Iss. 61, Vol. 16, No. 4, Dec. 2024 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.
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		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, CH₃ONa, and CH₃OK [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
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		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/γ-Al₂O₃ 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/γ-Al₂O₃ catalyst, with the main ingredient being duck egg shells, which are processed to produce the catalyst [13, 16].
Methodology: Experimental Design: The experimental design is presented clearly but lacks certain details that are crucial for reproducibility. Suggested Improvement: Provide more detailed specifications for equipment used, especially the	2. MATERIAL AND METODS 2.1 Meterials The materials used include duck egg shells, Barco brand coconut oil, methanol, NaOH, Gamma Alumina, and distilled water. 2 Methods 2.2.1 Preparation of CaO Catalyst from Egg Shells 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	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

microwave reactor. The conditions for calcination and catalyst preparation need clearer steps, including temperatures, times, and materials used. Microwave Parameters: While the reaction times and power levels are specified, more information is needed on how the microwave parameters were optimized. Suggested Improvement: Include a brief explanation of why certain power levels (400 watts) and reaction times were chosen and how these relate to other power levels used in the literature.	ground into a powder and passed through a 200-mesh sieve. Then the egg-shell powder is calcined in a furnace at a temperature of 900 °C for 6 hours until solid CaO is produced. 2.2.2 Impregnation of Egg Shells with NaOH and γ-Al₂O₃ Duck egg shell powder that has been calcined is impregnated using 1 gram of solid NaOH (pa) and 5 grams of γ-Al₂O₃ then 40 ml of distilled water is added. The mixture was stirred using a magnetic stirrer until it was homogeneous and became a paste. Place in the oven at 110 °C for 8 hours. The CaO/Na/γ-Al₂O₃ solid formed was heated at 500 °C for 3 hours. After that, it is ground again and sieved with a 200-mesh sieve. 2.2.3 Production of Biodiesel Coconut oil in as much as 50 ml for 30 minutes at a temperature of 60 °C. Then 24 ml of methanol and 1% CaO/Na/γ-Al₂O₃ catalyst were added to the coconut oil and stirred using a magnetic stirrer 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.	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-Al₂O₃ Duck egg shell powder that has been calcined is impregnated using 1 gram of solid NaOH (pa) and 5 grams of γ-Al₂O₃ 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-Al₂O₃ 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-Al₂O₃ 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.
Results and Discussion: Data Presentation: The results are well presented but can be expanded to include more discussion on the implications of the findings.	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 3.1. The test results show that CaCO₃ containing duck egg shells decompose into CaO after a six-hour heating process at 900 degrees Celsius. CaO production utilizes	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 CaCO₃ containing duck egg shells decompose into CaO after a six-hour heating process at 900 °C CaO production utilizes pulverized egg shells as the

Suggested Improvement: Discuss why the optimal biodiesel yield was observed at a catalyst concentration of 2.5% and the 7.5-minute reaction time, and what happens at higher concentrations and times. Characterization Data: Suggested Improvement: Compare the efficiency of the catalyst used in this study with other commonly used catalysts in the literature and discuss the advantages and limitations.	pulverized egg shells as the primary component, as illustrated in (Fig. 3.1.a) Egg shells have the same amount of CaO as the catalyst that is made when γ-Al₂O₃ and NaOH (pa) 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-Al₂O₃ functions as a catalyst support, which can improve the performance of a catalyst [10]. 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 a temperature of 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-Al₂O₃ catalyst product, shown in (Fig.3.1.b), is ready to be used to speed up the reaction. The assessment of catalyst activity was conducted on the transesterification process involving methanol and coconut oil for the production of biodiesel. This demonstrates that increasing the number of catalysts increases the number of 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 speed to increase [12]. It can be seen that from various variations in catalyst concentration, the smallest yield was obtained at a catalyst concentration of 1% and the	primary component, as illustrated in Figure 3. Egg shells have the same amount of CaO as the catalyst that is made when γ-Al₂O₃ 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-Al₂O₃ 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. 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%.
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	largest yield was obtained at a catalyst concentration of 2.5%.	
The paper includes characterization techniques like XRF and SEM, which are important. However, more explanation is needed about the SEM images and XRF results, especially on how they support the catalytic performance. Suggested Improvement: Add a more detailed explanation of how the morphology seen in the SEM images affects catalyst activity. Also, clarify the significance of the XRF analysis and relate it back to catalyst performance in biodiesel production.		
Catalyst Efficiency: The paper mentions that the use of CaO/Na/γ-Al₂O₃ can reduce reaction time and improve yield. However, there is little discussion of how this compares to existing catalysts used in	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 [13]. The power input to the microwave affects the wave amplitude. The greater the power used to generate microwaves, the greater the electric field produced	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

biodiesel production.	[14]. The rotation speed of polar molecules has a linear relationship to the microwave amplitude [15]. The longer the reaction time and the higher the temperature given, 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. This decrease will cause a reduction in the reaction between triglycerides and methanol, so the amount of biodiesel produced will decrease. The temperature has passed the boiling point of methanol, so some of the methanol undergoes a phase change from liquid to gas. This change in the methanol phase causes the amount of methanol in the liquid phase to decrease. Reducing the amount of methanol in the solution causes a reduction in the number of effective collisions to produce biodiesel, so the yield of biodiesel formed will decrease [1, 16]	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].
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Int Journal of IOTPE (IOTPE)

Balasan: Submission a new manuscript

Kepada: A. Suryanto

Dear Author,

Paper ID: 1901-240904

1. **Figure Quality (Figures 1, 2, 3, 5, 6, 8-15):** The figures still appear to be of low resolution, not redrawn using high-quality graphical tools. Many figures are still in formats like JPEG and PNG with low resolution, which could result in poor quality in the final published version.

2. **Redrawing Figures and Charts:** Figures and charts have not been redrawn using MS Word's Chart or Drawing tools as requested. They do not meet the high-fidelity quality requirement.

3. **Font and Size in Tables, Figures, and Algorithms:** There is inconsistency in font size and type used in tables, figures, and algorithms. They have not all been standardized to Times New Roman 8 pt, as requested.

4. **References Formatting:** Although some references have been revised, many do not completely follow the IOTPE journal style. Not all the references include details like volume, issue, page numbers, and correct punctuation. Additionally, it is unclear if at least two references from previous IOTPE journal issues have been included.

5. **Figure and Table References:** The paper still does not consistently refer to all figures, tables, and algorithms by their numbers in the text body.

6. **Equations and Parameters:** While the main equations seem to be formatted correctly using MathType, some parameters in the text that are not part of equations still do not consistently use the requested italic formatting.

7. **The 3rd Author Photo and Biography:** High-quality, color photographs and biography of the 3rd author are not included.

Summary:

The revised paper addresses some formatting and content requirements, such as using decimal points and improving equation formatting. However, key areas like figure quality, reference formatting, consistent font usage, and author photographs remain unaddressed or only partially corrected. Further revisions are needed to fully comply with the review comments.

Best regards,

Editor-in-Chief of International Journal on
"Technical and Physical Problems of Engineering" (IOTPE)
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A. Suryanto

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Kepada: Int Journal of IOTPE (IOTPE)

Dear Editor-In-Chief IOTPE

Here I attached the revised manuscript. Thank you

Best regards

Author

Lihat Lainnya dari Int Journal of IOTPE (IOTPE)



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Dari: Int Journal of JTPE (IJTPE) ijtpe0@gmail.com
Subjek: Re Submission a new manuscript
Tanggal: 5 Oktober 2024 15.38
Kepada: A. Suryanto a.suryanto@umi.ac.id



Dear Author,

Paper ID: 1901-240904

Please kindly revise your submitted paper considering the following Review Comments and resend it again for checking out.

REVIEW COMMENTS:

- Use the complete phrases of Figure instead of Fig. words.
- The quality of **Figure(s) 1, 2, 7** is not accepted and **should be changed or redrawn** by a high quality graphical tool(s). The quality of the figures in the journal published PDF format will be poor. Use the origin images with a qualitative resolution of **High Fidelity**.
- Extend, improve and reformulate CONCLUSION.

REFERENCES:

- References need to be re-written according to the JTPE Journal style as Name (first word) Surname, ..., (WITHOUT AND) "Paper or Book Title", Proceeding or Journal or Publication (COMPLETE NAME), Issue ..., Vol. ..., No. ..., pp. ...-..., Place, City, Country, Date/Year, *WITHOUT WEB ADDRESS, WITHOUT DOI*.

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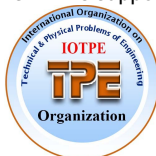
[1] **A.B. Cdef, G. Hijk, L.M.N. Opqr**, et al., "The Title of the Paper or Book or Thesis or Report", Conference of Abcd, Journal of Efgh, **Issue 1, Vol. 2, No. 3, pp. 4-10, City, Country, April 2021.**

- The maximum number of the references is 20-30. Please reduce the additional references.
- Use and address at least two references in your paper from the previously accepted papers in IJTPE journal issues published in the address, <http://www.iotpe.com/IJTPE/IJTPE-JournalIssues.html>.

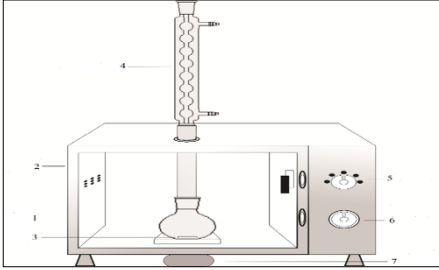
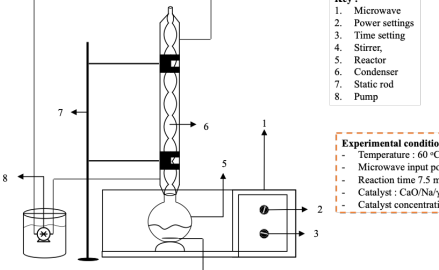
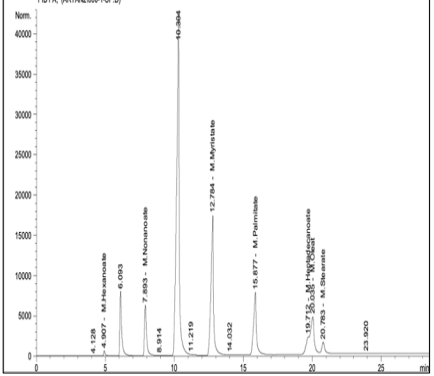
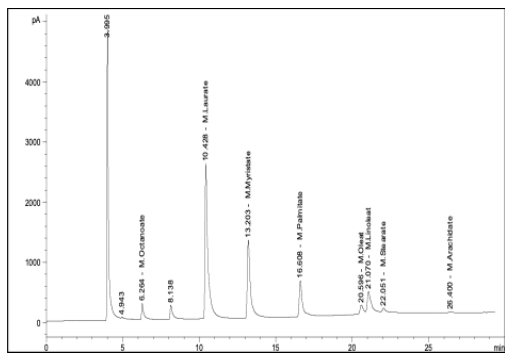
Best regards,

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Coments from Reviewers	Before Revision	Aftre Revision
Figure.1	 Fig.1 Schematic illustration of microwave reactor in situ transesterification; 1. Reactor, 2. Microwave, 3. Bar stir, 4. Condenser, 5. Power Setting, 6. Time setting, 7. Magnetic stirrer.	 Key : 1. Microwave 2. Power settings 3. Time setting 4. Stirrer 5. Reactor 6. Condenser 7. Static rod 8. Pump Experimental conditions - Temperature : 60 °C - Microwave input power, 400 W - Reaction time 7.5 min - Catalyst : CaO/Na/γ-Al₂O₃ - Catalyst concentration 1-3% Figure 1. Schematics illustration of the microwave reactor in situ transesterification
Figure.2	 Fig. 2 Egg shells, the basic material for producing catalysts: a. egg shell; b. CaO/Na/γ-Al₂O₃	 Fig. 2 Egg shells, the basic material for producing catalysts: a. egg shell; b. CaO/Na/γ-Al₂O₃
Figure 7.	 Fig.7 Chromatogram of Biodiesel product analysis Gas Chromatography	 Figure 7. Chromatogram of Biodiesel product analysis Gas Chromatography
Conclusion	The activity of the CaO/Na/γ-Al₂O₃ catalyst increases in the manufacture of biodiesel with the help of microwave heating. Microwaves can reduce the amount of catalyst and speed up 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 watts of microwave power; the resulting biodiesel product is 91.84 percent in volume and	In this research, the activity of the CaO/Na/γ-Al₂O₃ 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

	conforms to the SNI-7182 standard.	400 W of microwave power, the resulting biodiesel product is 91.84 percent in volume, with density quality specifications of 8.74 gr/cm ³ and conforms to the SNI-7182 product standard. Thus, this study becomes the basis for reducing global carbon emissions by green energy.
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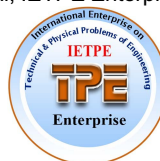
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MICROWAVE-ASSISTED TRANSESTERIFICATION FOR BIODIESEL PRODUCTION: CATALYST ACTIVITY OF $\text{Ca/Na}/\gamma\text{-Al}_2\text{O}_3$ DERIVED FROM EGG SHELLS

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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 $\text{CaO/Na}/\gamma\text{-Al}_2\text{O}_3$ 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.

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 alkalinized 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, $\text{CaO}/\text{Na}/\gamma\text{-Al}_2\text{O}_3$ 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 $\text{CaO}/\text{Na}/\gamma\text{-Al}_2\text{O}_3$ catalyst, with the main ingredient being duck egg shells, which are processed to produce the catalyst [13, 16].

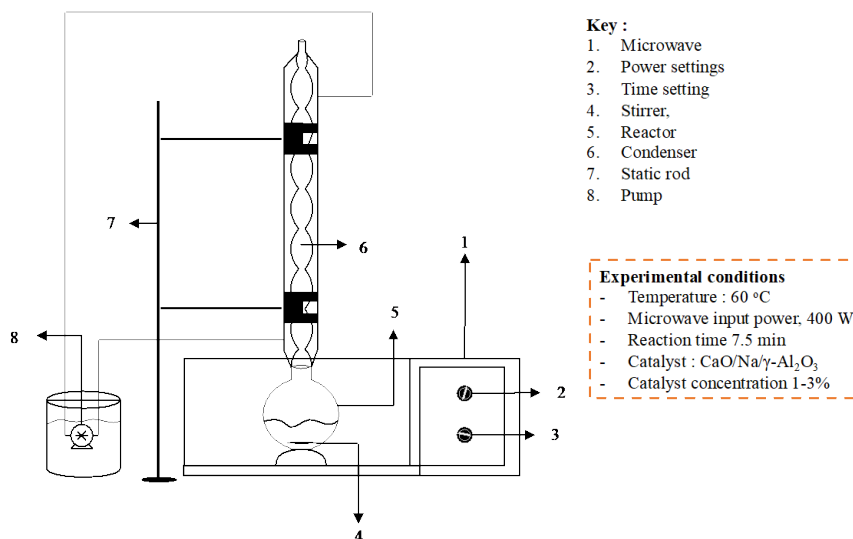


Figure 1. Schematics illustration of the microwave reactor in situ transesterification

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 -Al₂O₃

Duck egg shell powder that has been calcined is impregnated using 1 gram of solid NaOH (pa) and 5 grams of γ -Al₂O₃ 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 -Al₂O₃ 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 -Al₂O₃ 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 CaCO₃ 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 -Al₂O₃ 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 -Al₂O₃ 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 -Al₂O₃ 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 -Al₂O₃

3.2. X-Ray Fluorescence Analysis

The CaO/Na/ γ -Al₂O₃ 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 -Al₂O₃ catalyst using XRF

Parameter	Rate (%)
SiO ₂	0.07
Al ₂ O ₃	28.65
TiO ₂	0.02
Fe ₂ O ₃	< 0.001
CaO	52.83
MgO	0.37
K ₂ O	0.04
Na ₂ O	1.52
Na	1.12

The analysis results show that the elements Na and CaO are well distributed into the γ -Al₂O₃ 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 -Al₂O₃, 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 -Al₂O₃ 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.

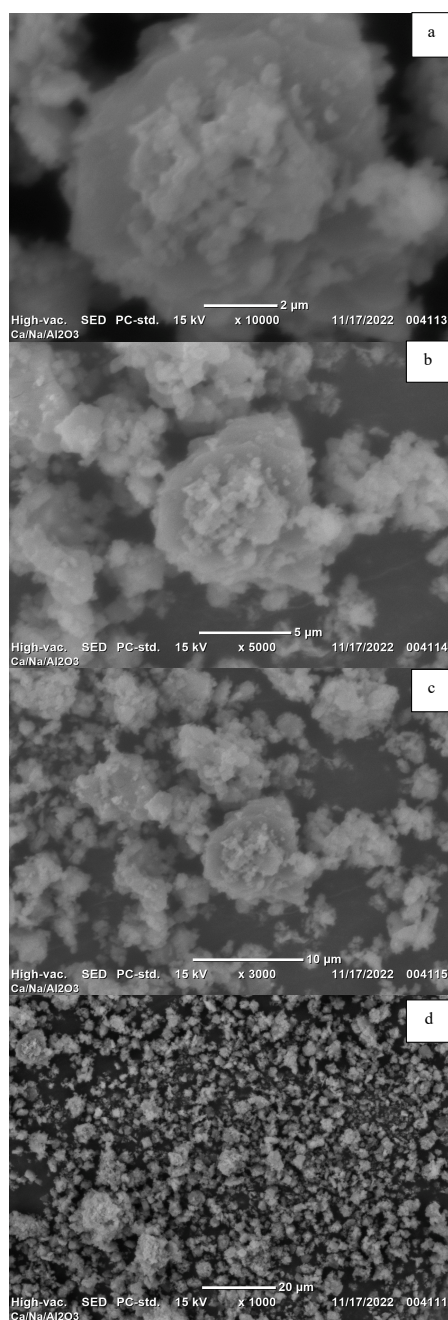


Figure 3. Catalyst Surface Morphology, a) 2000x magnification, b) 5000x magnification, c) 10000x magnification, d) 20000x magnification

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 $\text{CaO}/\text{Na}/\gamma\text{-Al}_2\text{O}_3$ 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 $\text{CaO}/\text{Na}/\gamma\text{-Al}_2\text{O}_3$ catalyst contains 74.05% CaO compounds, of which the Ca element is 58.92 and the $\gamma\text{-Al}_2\text{O}_3$ 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. $\text{CaO}/\text{Na}/\gamma\text{-Al}_2\text{O}_3$ Catalyst Activation Test with Biodiesel Production

The $\text{CaO}/\text{Na}/\gamma\text{-Al}_2\text{O}_3$ 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 $\text{CaO}/\text{Na}/\gamma\text{-Al}_2\text{O}_3$ 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.

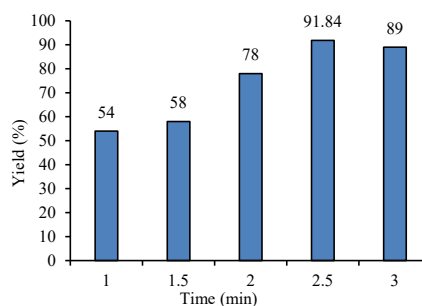


Figure 4. Effect of catalyst concentration on the production of methyl ester products

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.

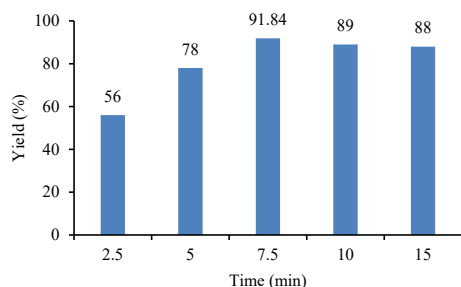


Figure 5. The relationship between the effect of reaction time on the yield of biodiesel products

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-Al₂O₃ 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.

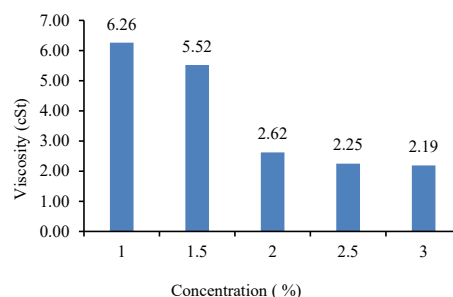


Figure 6. The Effect of catalyst concentration on the viscosity of biodiesel products at 400 W

According to Figure 6, the utilization of the CaO/Na/γ-Al₂O₃ 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].

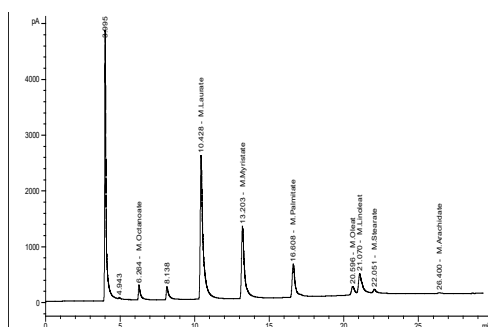


Figure 7. Chromatogram of Biodiesel product analysis Gas Chromatography

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

Parameters	Product	SN1-7182
Density (40 °C), g/ml	0.874	0.85-0.89
Kinematic Viscosity (40 °C), cSt	2.62	2-6
Acid Number, mg-KOH/g	0.38	max 0.4
Cetane Number, --	61	min 51
Flash Point, °C	135	min 130

4. CONCLUSIONS

In this research, the activity of the $\text{CaO}/\text{Na}/\gamma\text{-Al}_2\text{O}_3$ 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 SN1-7182 product standard. Thus, this study becomes basis for reducing global carbon emissions by green energy.

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