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Modification of CaO Catalyst from Chicken Eggshell with Wet Impregnation Method for Two-Stage Process of Biodiesel from Tamanu Oil

Kanesya Najah Abidin[1], Reza Gymnastiar[1], A.R. Yelvia Suniarti[1], Dyah Suci Perwitasari[1], Firman Kurniawanysah[2]

[1]Chemical Engineering Department, Universitas Pembangunan Nasional "Veteran" Jawa Timur, Surabaya
[2]Chemical Engineering Department, Institut Teknologi Sepuluh Nopember, Surabaya
*Corresponding author: kanesyanajah@gmail.com

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Abstract
Egg production from laying hens in Indonesia is so high that if the egg shells produced are not used properly, they will produce waste. Chicken eggshell waste has a high mineral content, one of which is calcium. Calcium can be utilized as a heterogeneous catalyst which can be used in making renewable energy such as biodiesel. Biodiesel is an alternative energy that is being widely developed because it can be an environmentally friendly alternative to diesel engine fuel. This research aims to determine biodiesel obtained from tamanu oil (Calophyllum inophyllum) by etherification and transesterification processes using a catalyst modified from egg shells using a wet impregnation method using alkaline potassium hydroxide. The catalyst used is the catalyst with the highest alkalinity for making biodiesel. The biodiesel produced had the highest yield of 76.9040% with a density value of 0.94981 gr/ml and a viscosity of 4.11412 cSt.
Keywords: *chicken egg shells, impregnation, tamanu, esterification, transesterification*

Abstrak
Produksi telur ayam petelur di Indonesia sangatlah tinggi sehingga cangkang telur ayam yang dihasilkan nantinya apabila tidak dimanfaatkan dengan baik akan menghasilkan limbah. Limbah cangkang telur ayam memiliki kandungan mineral yang tinggi salah satunya kalsium. Kalsium dapat dimanfaatkan menjadi katalis heterogen yang dapat digunakan dalam pembuatan energi terbarukan seperti halnya biodiesel. Biodiesel menjadi salah satu energi alternatif yang sedang banyak dikembangkan karena dapat menjadi alternatif pengganti bahan bakar mesin diesel yang ramah lingkungan. Penelitian ini bertujuan untuk mengetahui biodiesel yang diperoleh dari minyak nyamplung (*Calophyllum inophyllum*) dengan proses eterifikasi dan transesterifikasi menggunakan katalis yang dimodifikasi dari cangkang telur dengan menggunakan metode impregnasi basah menggunakan alkali kalium hidroksida. Katalis yang digunakan yaitu katalis dengan kebasaan tertinggi untuk pembuatan biodiesel. Biodiesel yang dihasilkan memiliki yield terbanyak sebesar 76.9040% dengan nilai densitas sebesar 0.94981 gr/ml dan viskositas sebesar 4.11412 cSt.
Kata Kunci: *cangkang telur ayam, impregnasi, tamanu, esterifikasi, transesterifikasi*

1. Introduction

According to data obtained from the Central Statistics Agency, in 2022 egg production from laying hens in East Java province will reach 1,314,114.93 tonnes. Meanwhile, the quality of chicken eggs in Indonesia according to Qurniawan et al., 2022 has an average weight of 57.6 grams with a shell weight of 7.3 grams or 12.39% of the egg weight. So, if calculated, every year 162,818.8398 tonnes of chicken egg shells can be produced and if they are not utilized they will become waste which will accumulate over time. Chicken egg shells can be a natural source of minerals because chicken egg shells contain many minerals such as Ca, Mg, Zn and Cu. According to Schaafsma et al., 2014 calcium (Ca) is the highest mineral content in chicken egg shells, namely 40.1% compared to other minerals such as Mg at 0.45% and Zn at 0.000513%. The calcium in egg shells is available in the form of carbonate. Where, Calcium Carbonate can decompose into Calcium Oxide when calcined at temperatures above 800°C [3].

A catalyst is a component that can help increase the rate of a chemical reaction without reacting in the process. Catalysts exhibit a different pathway that requires a lower supply of activation energy [12]. Because the activation energy required in the reaction is lower, the time required to achieve the reaction is also faster. Making biodiesel generally takes a long time, so a catalyst is needed to speed up the synthesis process [5]. Chemical catalysts have two types that differ according to their phase, namely homogeneous and heterogeneous catalysts. Homogeneous catalysts have the same phase as the reactant phases such as NaOH, KOH, and H2SO4. Meanwhile, heterogeneous catalysts have a phase that is different from the reactant phase, which is widely available in solid form, such as CaO, BaO, and SrO.

The CaO catalyst is one of the most researched catalysts for biodiesel production because of its high activity, abundant availability and low cost [8]. Apart from that, CaO is also a catalyst that has a fairly high base value. The CaO catalyst is in solid or heterogeneous form, making it easier to separate the biodiesel product [1]. There have been several studies regarding the use of CaO as a catalyst for the biodiesel production process, but the results obtained have not been optimal so research into catalyst development is still ongoing. One of the problems is that the CaO catalyst is very reactive to water, causing the formation of Ca(OH)2 which can reduce the activity of the catalytic reaction. So it is necessary to carry out innovations, one of which is the process of inserting metal into CaO through a wet impregnation process, where this can affect the active side of the catalyst and can increase the surface area [10].

Several previous studies, such as those carried out by [1], on the reaction of making biodiesel using a CaO catalyst from CaCO3 material, can produce a biodiesel yield of 98.8%. Apart from that, in experiments on biodiesel production from soybean oil using a 4% CaO/KOH catalyst (15% KOH impregnation), the biodiesel product yield was 97.1% [15]. Therefore, this research aims to develop a CaO-KOH catalyst from chicken eggshell waste. Chicken egg shells are a source of CaO with metal inserts, namely KOH, at several concentration variations through a wet impregnation process. The best catalyst from the analysis results will be used as a catalyst in the biodiesel catalytic reaction through the Two Stage Process using tamanu oil.

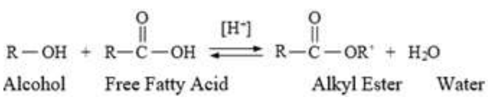
Tamanu (Calophyllum inophyllum L.) in various regions is known by different names. In England it is known as Tamanu, in China it is known as Hai-Tang-Guo, and in Indonesia, it is known as nyamplung. Nyamplung is a tropical plant that grows in the plains and coastal areas. In various Asian countries, this plant is used for medical and cosmetic purposes [6]. Nyamplung plants have fruit that can be used every year because on average each year per tree can produce 250 kg of seeds [9]. These seeds have a high oil content, which is around 60% in fresh seeds and 75% in dry seeds [11]. Biodiesel is a fuel consisting of a mixture of mono-alkyl esters of long-chain fatty acids, which is used as an alternative to fuel from diesel engines and is made from renewable sources such as vegetable oil or animal fat [13]. Biodiesel is made through a chemical process called esterification or transesterification. Biodiesel is used because it is biodegradable, non-toxic, and has low emissions [9]. Apart from that, the advantage of biodiesel is that it produces lower exhaust emissions, both in terms of particulate matter (PM), total hydrocarbons (THC), and carbon monoxide (CO). The biodiesel products produced must meet the quality standards and standards set by the Indonesian National Standards Agency. The following Biodiesel Standards and Quality based on SNI are explained in **Table 1**.

Table 1. Biodiesel Standards and Quality SNI 7182:2015

Parameter	Unit. Min/max	Requirements
Density at 40 °C	kg/m3	850-890
Kinematic Viscosity at 40 °C	mm2/s (cSt)	2,3-6,0
Acid Number	mg-KOH/kg, max	0,4

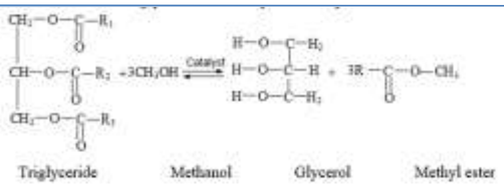
Source : BSN (2017)

The biodiesel synthesis process can go through the esterification stage and continue with the transesterification stage. Before going through the esterification process, the oil raw materials used must be free from water content. This is intended to avoid saponification reactions.



(1)

Esterification aims to reduce FFA levels in oil. The FFA requirement for oil raw materials to be used in the transesterification process is <2%. So, oil raw materials need to be esterified by reacting with an acid catalyst. This esterification reaction is the initial reaction for the conversion of FFA into methyl ester. The catalytic reaction of biodiesel uses a heterogeneous catalyst, namely CaO, which is used in the transesterification reaction to convert triglycerides into fatty acid methyl ester.



(2)

2. Material and Methods

Material

The materials used in the research include tamanu seed oil obtained from Karanganyar, Central Java, egg shells from laying hens from food business waste in Surabaya, East Java, Sulfuric Acid 98% (CIMS-INDO), Methanol 99% (CIMS-INDO) , Aquades, Potassium Hydroxide ProAnalis (Merck), Oxalic Acid (CIMS-INDO), Phenolphthalein (CIMS-INDO), Ethanol 96% (CIMS-INDO), Benzoic Acid (SAP), Ethanol 99% (SmartLab)

Instrumentation

The tools used in the research include an oven, furnace, glass beaker, magnetic stirrer, measuring cup, Kara funnel, filter paper, 100 mesh sieve, stirring rod, dropper pipette, Erlenmeyer, porcelain cup, series of calcination tools, and series of batch reactor tools. Meanwhile, the tools used in data analysis include the PANalytical XRF tool, the SEM-EDX FEI Inspect S50 tool, statives, clamps, burettes, Ostwald viscometers, pycnometers, and thermometers.

Procedure

1) Preparation of catalyst raw materials

   Chicken egg shells are washed to remove dirt and white membranes until clean and then reduced in size. The chicken egg shells are then dried in the oven at 100℃ for 1 hour. The dried chicken egg shells are then size reduced to 100 mesh and become chicken eggshell powder.

2) Modification of catalysts

   a. Modification of catalysts using KOH impregnation method

      The chicken eggshell powder is calcined at a temperature of 900℃ for 3 hours to decompose CaCO3 of the chicken eggshell into CaO. CaO is then impregnated with KOH solution. The method is to dissolve 50 grams of CaO in 200 ml of KOH solution (0, 3, 5, 10, 12, 15% w/v) and stir using a magnetic stirrer for 1 hour until it forms a slurry. The slurry is filtered with filter paper to reduce the liquid in the slurry thereby speeding up the drying process. The filtration precipitate was dried in an oven at 100℃ for 24 hours. Then, the results of drying in the oven are size reduced to 100 mesh. The material is continued with the second calcination at a temperature of 450℃ for 4 hours so that KOH can be decomposed into K2O and K2CO3 which can occur at temperatures above 427℃ to form a CaO-KOH catalyst.

   b. Alkalinity Test

      To measure the basicity of a solid catalyst, a Hammett indicator is used using the acid-base titration principle. Where titration is used with a benzoic acid solution of 0.02 mol/l anhydrous ethanol. The catalyst sample was dissolved in 50 ml of distilled water, then stirred with a magnetic stirrer for 1 hour and filtered using filter paper. The filtrate obtained was then dripped with phenolphthalein indicator and titrated with a benzoic acid solution of 0.02 mol/l anhydrous ethanol until the pink color disappeared. Basicity is determined in mmol/g using the formula:

$$\text{Alkalinity}\left(\frac{\text{mmol}}{\text{gr}}\right) = \frac{(V_{\text{benzoic acid}} \times N_{\text{benzoic acid}})}{m_{\text{catalyst}}} \qquad (3)$$

3) Biodiesel production

   a. Esterification Process

      Nyamplung oil is esterified first by reacting with methanol with a mole ratio of oil: methanol of 1:18 and sulfuric acid 1% v/v oil at a temperature of 65℃ for 1 hour. The esterified oil was put into a separating funnel and left for 12 hours. The oil in a separating funnel is separated from the by-products. The esterified oil is tested for free fatty acid (ALB) content first using alkalimetric titration until it meets SNI (FFA<2%).

   b. Free Fatty Acids Level Test

      Analysis of Free Fatty Acid (ALB) levels is carried out by dissolving the oil in ethanol which is neutralized with KOH then adding phenolphthalein indicator and titrating with standard KOH until it reaches the pink end point of titration (TAT) which does not disappear, then the calculation is carried out using the following formula:

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$$\text{Acid Number}\left(\frac{mg\ KOH}{g\ sampel}\right) = \frac{(A-B)\times N\times 56.1}{W} \quad (4)$$

Notes:
A = KOH volume of sample titration (ml)
B = volume of KOH blank titration (ml)
N = standard KOH concentration (N)
W = sample weight (mg)

Where when %FFA is stated, the acid number is divided by 1.99 for oleic; 2.81 for laurate, and 2.19 for palmitate. If the free fatty acid content is <2%, it can be continued in the process of making transesterification biodiesel

c. Transesterification Process

50 ml of esterified oil was transesterified with a mole ratio of oil: methanol of 1:9 and a CaO catalyst impregnated with 10% KOH with varying weight percentages, namely 2%, 3%, 4%, 5%, and 6% w/v in total. temperature 65°C for 3 hours. The oil resulting from transesterification is then put into a separating funnel and left for 12 hours before being separated between biodiesel and by-products in the form of solvents and catalysts. The result of transesterification is biodiesel which still contains glycerol. Hence, it needs to be separated by washing using distilled water at a temperature of 50°C repeatedly until glycerol is no longer formed or the color of the washed distilled water is clear again. Glycerol-free biodiesel that has been washed is then oven at 105°C for 1 hour. Biodiesel products free of by-products are analyzed for density, viscosity, ALB content, and yield value

3. Results and Discussion

Component of Chicken Eggshell

The raw material for chicken egg shells used as a CaO catalyst was subjected to an initial raw material test to determine the element content in chicken egg shells using the XRF analysis. The figure shows a graph of peak formation for each element resulting from qualitative XRF characterization. The XRF characterization pattern graph qualitatively explains the various peak changes. Where the Ca element shows a peak with the highest intensity among the other elements.

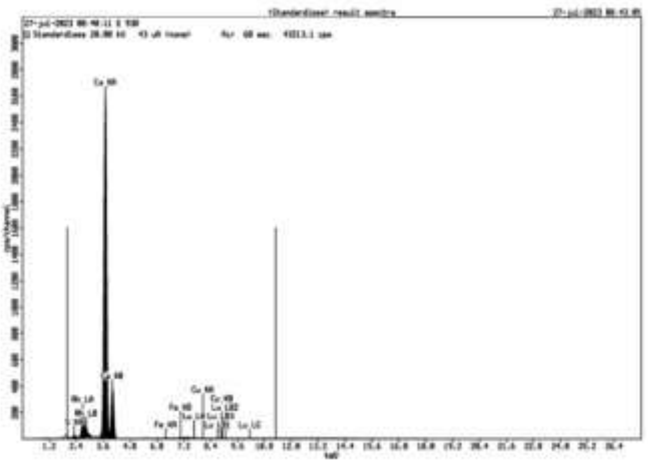

Fig. 1 : XRD Result of Chicken Eggshell

Table 2. Elemental analysis of chicken eggshells

Element	%
S	0,35
Ca	99,29
Fe	0,095
Cu	0,041
Lu	0,22

Meanwhile, the table shows the results of quantitative XRF characterization where chicken egg shells contain the elements S, Ca, Fe, Cu, and Lu. The highest element content is calcium (Ca) with a percentage of 99.29% followed by a percentage of other compounds which is less than 0.7%. According to [7], the results of XRF analysis show that the compound obtained from the calcination of chicken egg shells at a temperature of 1000 °C contains 99.48% of the element Ca. The high calcium content in egg shells can be used as a heterogeneous catalyst, however, the calcium in chicken egg shells is still in the form of calcium carbonate (CaCO3) and needs to be decomposed to become CaO.

Alkalinity of Modified Catalyst

The Hammett basicity test was carried out to determine the basicity of the modified catalyst using the wet impregnation method with KOH in units of mmol/gram of benzoic acid. Impregnation is carried out to increase the basicity of the CaO catalyst by inserting KOH metal. This is because CaO tends to bind water in the air so that it can reduce its catalytic activity, therefore it is necessary to increase the alkalinity of the catalyst so that it can work as a catalyst for the transesterification process optimally. **Figure 2** shows that the higher the concentration of KOH impregnated into the catalyst, the more basic the catalyst is. The lowest alkalinity was obtained at a KOH impregnation concentration of 0%, namely 1.59 mmol/g benzoic acid.

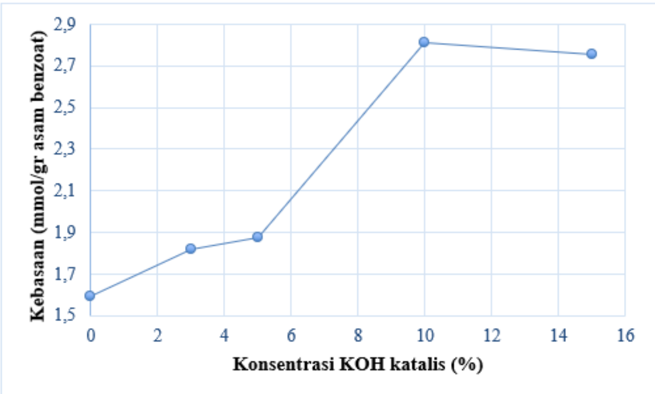

Fig. 2: Alkalinity Test Results

Meanwhile, the highest alkalinity was obtained at a KOH impregnation concentration of 10%, namely 2.98 mmol/g benzoic acid. However, there was a decrease in the alkalinity value of the catalyst at a KOH impregnation concentration of 15% to 2.753 mmol/g benzoic acid. According to (Oko & Kurniawan, 2019) a decrease in the basicity of the impregnated catalyst occurs because the catalyst is already in a supersaturated state so it can no longer accept KOH into its pores. In addition, because during the impregnation process, there is stirring, this can cause KOH which has been inserted into the CaO pores in a supersaturated state to experience desorption and ultimately reduce its basicity value.

Morphology of Catalyst

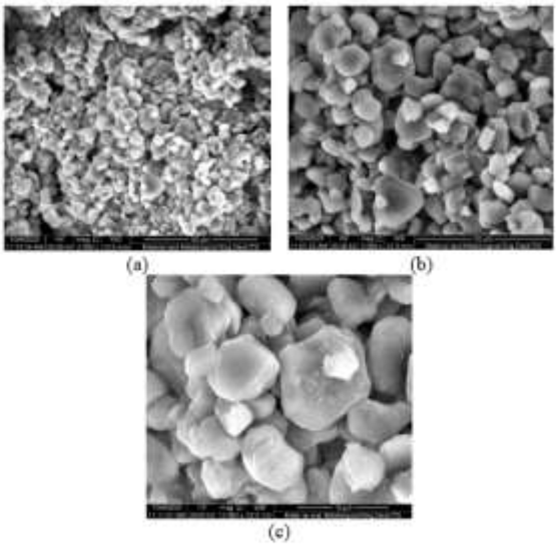

Fig. 3: SEM analysis results of impregnated catalyst 10%KOH (a) 2500× (b) 5000× (c) 10000×

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The morphology and composition of the catalyst can be determined by SEM-EDX analysis. The catalyst used is a catalyst with the highest alkalinity, namely a catalyst with a KOH impregnation concentration of 10%. Through 10000× magnification, it can be seen that the catalyst still tends to be CaO with KOH visible inserted in several parts. This is the result of the KOH impregnation method that has been carried out.

Table 3. EDX Results in Component Composition of Impregnated Catalyst 10% KOH

Element	Composition (Wt%)
CK	3,84
OK	33,27
KK	10,04
CaK	52,85

The known catalyst composition as in **Table 3** shows that calcium is the compound with the highest content, followed by oxygen and potassium compounds. The oxygen contained has a high composition because the compounds used both have oxygen groups, namely Calcium Oxide (CaO) and Potassium Hydroxide (KOH). So it can be seen that the wet impregnation method that has been carried out has succeeded in inserting potassium metal into the CaO catalyst.

Biodiesel Characterization

The process of making biodiesel is carried out using 2 stages, namely esterification and transesterification. The esterification process is carried out to reduce the level of free fatty acids contained in the raw material oil, namely tamanu oil, using the strong acid catalyst H2SO4 until it meets SNI standards, namely <2%. This is done so that later a saponification reaction does not occur in the transesterification process which uses a base catalyst that can result in the formation of soap and reduce the biodiesel yield obtained [14]. In the esterification reaction between tamanu oil and methanol, the ALB content was obtained at 1.993%, where this value decreased from the previous value of 19.4522%. With a free fatty acid content of less than 2%, the oil resulting from the esterification process can be used in the transesterification process.

Free Fatty Acid

Biodiesel from tamanu seed oil with a catalyst that has the highest alkalinity, namely CaO/KOH 10% with a weight percentage of 2%, 3%, 4%, 5%, and 6% has an acid value of 0.1807; 0.0947; 0.3098; 0.2409; and 0.2409 (mg-KOH/g). Meanwhile, based on SNI parameters, it shows that biodiesel has an acid number with a maximum value of 0.4 (mg-KOH/g). From the results of this analysis, it can be seen that biodiesel from Nyamplung oil meets the requirements. The acid number of the biodiesel produced can affect the corrosion performance of the engine when using this fuel so that the acid number of biodiesel is not more than 0.4.

Table 4. Free Fatty Acid of Biodiesel

Catalyst Concentration (%Wt/v)	Free Fatty Acid (FFA%)	Acid Number (mg-koh/g)
2%	0,9098	0,1807
3%	0,4765	0,0947
4%	1,5596	0,3098
5%	1,2130	0,2409
6%	1,2130	0,2409

Density

Biodiesel formed from tamanu oil through a transesterification process with a catalyst that has the highest alkalinity, namely CaO/KOH 10%, is analyzed for density to determine the biodiesel standard by SNI. From the results of the density analysis, it can be seen that biodiesel at the highest biodiesel yield of 6% Wt/v has a density of 949.81 kg/m3. Meanwhile, the SNI parameters state that biodiesel has a density in the range of 850 – 890 kg/m3. From these results, the biodiesel density at the highest yield does not meet the requirements. Apart from that, if we look at other weight percent values, the density is also not sufficient. This can happen because the biodiesel obtained still contains other compounds and during the separation process the by-products were not separated properly, so the density value obtained still does not meet SNI biodiesel standards. Density is related to the heating value and power produced by a diesel engine. Low density will produce a high heating value and vice versa [2].

Table 5. Density of Biodiesel

Catalyst Concentration (%Wt/v)	Density (gr/ml)
2%	0,94579
3%	0,90785
4%	0,94121
5%	0,94316
6%	0,94981

Viscosity

Biodiesel from tamanu seed oil with a catalyst that has the highest alkalinity, namely CaO/KOH 10% with a weight percentage of 2%, 3%, 4%, 5%, and 6% has a kinematic viscosity of 5.3751 cSt; 5.2337 cSt; 5.2864 cSt; 5.5458 cSt; and 4.1142 cSt. Meanwhile, based on SNI parameters, it shows that biodiesel has a viscosity in the range of 2.3 – 6 cSt. From the results of this analysis, it can be seen that biodiesel from Nyamplung oil meets the requirements. In the fuel injection process, viscosity has an important role. A viscosity value that is too low can result in leaks in the fuel injection pump. Meanwhile, a viscosity that is too high can cause the fuel to atomize into large droplets that have high momentum and can collide with the cylinder walls so that the injection pump cannot atomize properly.

Table 6. Viscosity of Biodiesel

Catalyst Concentration (%Wt/v)	Kinematic Viscosity (cSt)
2%	5,3751
3%	5,2339
4%	5,2864
5%	5,5458
6%	4,1142

Yield

The transesterification process is carried out using a catalyst with the highest basicity, namely CaO/KOH 10%. The number of catalysts used was varied to determine the optimum catalyst concentration in producing this Nyamplung oil biodiesel yield.

Table 7. Biodiesel Yield

Catalyst Concentration (%Wt/v)	Kinematic Viscosity (cSt)
2%	52,9704
3%	53,3480
4%	61,5789
5%	50,4595
6%	76,9040

From **Table 7** it is known that the largest yield obtained was 76.904% with a catalyst concentration of 6% Wt/v. The percent yield obtained tends to increase as the catalyst concentration contained in the transesterification process increases. This happens because a high amount of catalyst can speed up the reaction and produce higher biodiesel yields [5]. So the higher the catalyst concentration at the same reaction time, the faster the resulting reaction rate will be because the amount of catalyst used is higher so that it can produce more biodiesel yields. However, in the research, there was a decrease in the yield obtained at a catalyst concentration of 5% Wt/v which caused inconsistencies because at a concentration of 6% Wt/v, the yield obtained increased again.

This can happen because the operating temperature is less controlled, resulting in the operating temperature being used exceeding the optimal temperature. According to [12], if the temperature used exceeds the optimal temperature range required, it will result in a decrease in biodiesel yield because the triglyceride saponification reaction occurs well at high temperatures.

4. Conclusion

Based on the results of research conducted from the characteristics of raw materials for catalysts to the application of catalysts in making Nyamplung oil biodiesel, it was found that the catalyst with the highest alkalinity was obtained at a KOH impregnation concentration of 10%, namely 2.98 mmol/g of benzoic acid. The impregnation concentration was applied to make biodiesel and the weight of the catalyst so that a maximum yield value of 76.9040% was obtained at a weight of 6%Wt/v. It was also found that the viscosity value was 4.1142 cSt and the acid number was 0.2409 mg-KOH/g which met the SNI quality standards for biodiesel.

5. Acknowledgment

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