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Biodiesel Production From *Calophyllum Inophyllum* Using Base Lewis Catalyst

Lailatul Qadariyah* *[*,1]
***Donny Satria Bhuana* [1]**
***Raka Selaksa* [1]**
***Ja'far As Shodiq* [1]**
***Mahfud Mahfud* [1]**

[1] *Department of Chemical Engineering, Faculty of Industrial Technology, Institut Teknologi Sepuluh Nopember. Kampus ITS Sukolilo Surabaya 60111, Indonesia*
[*]*e-mail:* lqadariyah@chem-eng.its.ac.id

The search for renewable alternative energy must be developed, one of which is biodiesel. Seed *Calophyllum inophyllum* has a fairly high oil content of about 71.4% by weight, has great potential when used as raw material for making biodiesel. The purpose of this research was to synthesize biodiesel from *Calophyllum inophyllum* oil through the transesterification process using base lewis catalyst with microwave assisted, comparing the performance of sodium acetate to potassium hydroxide, knowing the amount of catalyst required to obtain the best biodiesel, and knowing the optimum power in the manufacture of biodiesel, The first step of making *Calophyllum inophyllum* biodiesel is degumming process, then continued with esterification. The next process is transesterification, followed by purification of biodiesel. The catalysts used are CH_3COONa, and KOH. From the results, *Calophyllum inophyllum* oil can be used as biodiesel feedstock, the best operating conditions for base catalyst at 300 W power, 1% (w/w) concentration, produced the best yield is 96% (for KOH catalyst) and 87% (for CH_3COONa catalyst).

Keywords : Biodiesel, *Calophyllum inophyllum*, Microwave, Base Lewis Catalyst

INTRODUCTION

Based on Indonesia Energy Outlook (Nugraha, 2016), final energy consumption during 2000-2014 is still dominated by BBM (petroleum, petrosolar oil, kerosene, fuel oil and aviation fuel). In 2000, petrosolar oil consumption had the largest share (38.7%) followed by kerosene (23.4%), gasoline (23.1%), fuel oil (9.6%) and avtur (5.2%). Furthermore, in 2014 to be gasoline (45.5%), petrosolar oil (45.2%), avtur (6.3%), and kerosene and fuel oil respectively by 1.5%. From these data, it appears that the use of fuel tends to increase from 2000-2014 (except kerosene and fuel oil) and by 2014, petrosolar and gasoline consumption is higher than other fuel. Increased consumption of fuel, especially gasoline and diesel fuel is mainly due to the number of vehicles that each year has increased. In general the reserves and age of national fossil energy resources are shown by **Table 1**.

Table 1. Reserve and Age of National Energy Sources (British Petroleum, 2017)

Energy Type	Avaibility	Remaining age (year)
Crude Oil	4,7 billionbarrel	15
Natural Gas	9,3 TSCF	35
Coal	4,968 billion ton	61

Given the declining availability of petroleum and the increasing rate of national oil consumption, an alternative renewable energy source is needed to achieve national energy security, one of which is biodiesel (Alamsyah et al., 2011).

Biodiesel can be produced using vegetable oil derived from seeds *Calophyllum inophyllum* plant. Some of the advantages of biodiesel produced from *Calpohyllum inophyllum* contain oil relatively high compared to other types of plants (jatropha 40-60%, Sawit 46-54%, and *Calophyllum inophyllum* 40-73%). The most compositions in nyamplung oil are oleic acid (39%) and linoleic acid(31%). *Calophylum inophyllum* seed oil is a potential renewable energy resource as a material of biodiesel without having to compete with food needs (Puspitahati et al., 2011).

However, production of biodiesel from these sources encountered many challenges such as high capital cost and limited availability of fat and oil resources. Biodiesel itself is a new energy source considering the manufacturing process must emphasize the effectiveness of energy use (Wati et al., 2011). Microwave radiation can be used to accelerate the reaction because the energy is directly transferred to the reactant, therefore the heat transfer process is more effective when compared to conventional heating and need shorter time to complete the reaction (Kirubakaran and Arul Mozhi Selvan, 2018). Eventually, the use of microwave is the best method to reduce reaction time and produce greater yield on biodiesel production (Motasemi and Ani, 2012).

Biodiesel obtained compared to biodiesel quality standards according to Indonesian National Standard (SNI 04-7182-2006). The test includes the density, viscosity, and yield of biodiesel.

This research is aim to give information about product quality of biodiesel from *Callophylum inophyllum* as new potential feedstock using sodium acetate and to compare the product quality with an alkaline catalyst such as potassium hydroxide.

METHODS

Material

Calohyllum inophyllum oil was commercially available from Cilacap area of Central Java and receive in liquid form extracted using mechanical press the oil that comes out of the press machine is dark green, which had high viscosity oil (54.13 cSt) and had a sufficiently high acid number, ie 58.20 mg KOH/g of oil. an one neck flash with an inner volume 1000 ml. Using reflux condenser as cooler tower. Modified Microwave (Electrolux Co., model EMM 2308X) as energy source equipped with magnetic stirrer.

Pre-treatment

It was necessary to have some pre-treatment before transesterification. This pre-treatment stage consists of degumming which aims to remove gum which consists of phosphatide, impurities and protein and esterification stage which serves to convert free fatty acid (FFA) to methyl ester and water so that it can be continued to transesterification stage.

100 ml of *Calophylum inophylum* oil were loaded and heated at a temperature of 80 ^{0}C then followed by the addition of 20% phosphoric acid by 0.3% (w/w). Once loaded, the reactor was sealed tightly. *Calophyllum inophyllum* oil were mixed for 15 minutes using magnetic stirer. The collected product was washed three times, each using warm aquadest (60°C), in a separation funnel. The upper oil phase was withdrawn and dried using an oven at 110°C to reduce the water content in the oil.

10 ml of post-degumming oil were loaded to reactor. Methanol was added to reactor alongside with the oil, the mole ratio of oil-methanol 1:40 and 98% H_2SO_4 catalyst by 13% (v/v). The reaction were carried out with constant stirring (~300 rpm) at 150 W microwave oven for 60 minutes. After the radiation process, separation between methanol, oil and catalyst was using separating funnel, the top layer of methanol which can be recovered and the bottom layer was a mixture of oil and methyl ester (crude biodiesel). The solution was clarify, washed three times, each using warm aquades (60°C). The crude biodiesel product was dried in an oven at 110°C and result from esterification is analysed.

Transesterification Step

Mixed Crude biodiesel (10 ml) were loaded in a methanol (mole ratio of oil-methanol 1:9) and acid catalyst was added in reactor. The reaction were carried out with constant stirring using magnetic stirrer at radiation in microwave oven with variable power for 1 hour. The solution washed three times, each with warm aquadest (60 °C) using a separator funnel. The oil was dried in an oven at 110 °C to reduce water content. The variables used are lewis base catalyst type: (KOH), (CH_3COONa), and catalyst concentration (% (w/w) oil): 0,5%; 0,75%; 1%; As well as microwave power (W) of 150, 300, 450, and 600.

RESULT AND DISCUSSION

Degumming

Changes of *Calophyllum inophyllum* oil after degumming process represents in **Table 2**. Changes that occur are decreased viscosity, and density and discoloration. Changes that occur due to the separation of gums from oil.

Table 2. The characteristic of oil *Calophyllum inophyllum*

Parameter	Before Degumming	After Degumming
Density at 40 °C (g/ml)	0,948	0,9307
Viscosity at 40 °C (cSt)	54,13	45,7
Colour appreance	Dark Green	Reddish Brown

Esterification

Esterification was performed to convert the carboxylic acid to esters. The reaction also produces water but it can be overcome by using an excess of methanol. Water from esterification process will be dissolved in methanol so that it does not inhibit the reaction process (Pan et al., 2016). In this process, it is required to reduce FFA levels less than 2% of oil before entering transesterification step to avoid saponification (Sudradjat et al., 2010.). To determine the optimum esterification operation condition to obtain *Calophyllum inophyllum* oil with FFA level less than 2% (acid number less than 4 mg KOH/g of oil) need to consider (Trombettoni et al., 2017), such as mole ratio of oil-methanol, catalyst concentration and microwave power. Acid number of *Calophyllum inophyllum* oil was high (58.20 mg KOH/g oil). This research uses a fixed power of 150 watts for the esterification process. This was expected to run at 60 ^{0}C with 1 hour of reaction time (Atabani, 2011). The results for this process was mole ratio of 1:40 oil-methanol using concentration of 13% H_2SO_4 (v/v) as catalyst. The final acid number is 3,8424 mg KOH/g oil (Supriya B. Chavan et al., 2014), so can be continued to transesterification step. At this esterification stage, the content of triglycerides in the oil does not react to be methyl esters because they did not show the formation of glycerol. This represent that in oils with high FFA concentrations when present in acidic conditions, the reactions occurring tend to be between FFA and methanol (esterification) and not between triglycerides and methanol (transesterification) (Muhammad, et.al 2014).

Transesterification

Transesterification was perfomed to produce triglycerides or diglycerides in *Calophyllum inophyllum* oil with methanol) results in Fatty Acids Methyl Esters (FAME) or biodiesel and glycerol as side products. **Figure 2** shows the effect of power on biodiesel yield on each base lewis catalyst and catalyst concentration. The curva tends to increase from 150 W to 300 W and tends to 300 W to 600 W tends to decrease. It indicate microwave power has a strong influence in catalyst activity (Di Serio et al., 2005). Using an alkaline catalyst, the use of power above 300 W, will cause a saponification reaction. The transesterification of the base catalyst of the reaction takes place at room temperature. When without a catalyst, the reaction requires a minimum temperature of 250°C (Comyns, 2007).

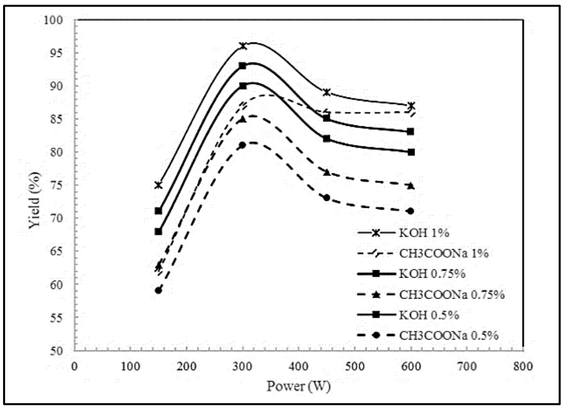

Fig. 2. Effect of microwave power on biodiesel yield

In addition **Figure 3** shows the influence of catalyst concentration. From the catalyst concentration of 0.5% to 1% the yield value tends to increase. The highest yield can be obtained using catalyst of KOH and at optimum power of 300 W, the increasing of catalyst concentration from 0.5% (w/w) to 0.75% (w/w) tend to increase the yield 3% (from 90% to 93%), and at the catalyst concentration from 0.75% (w/w) to 1% (w/w) increased yield by 3% (from 93% to 96%). In the catalyst of CH_3COONa and at optimum power of 300 W, the increasing of catalyst concentration from 0.5% (w/w) to 0.75% (w/w) increased yield 4% (from 81% to 85%), and in catalyst concentration from 0.75% (w/w) to 1% (w/w) increases yield by 2% (from 85% to 87%). In both KOH and CH_3COONa catalysts, yield increases from 0.5% (w/w) to 0.75% (w/w) concentrations are relatively significant, with relatively high yields at 1% catalyst concentration. The increase of catalyst concentration from 0.75% (w/w) to 1% (w/w) did not give result in a significant increase in yield of biodiesel, as well as all power variables, resulting in the best yield at 1% catalyst concentration (w/w). The high concentration of catalyst could increase of yield as the impact the presence of FAME (Abdullah et al., 2017). From the results of this study can be concluded that the base lewis catalyst with 1% (w/w) oil content is the most optimal catalyst level. In this case it can be expressed from **figure 3** the increasing the concentration of the base lewis catalyst the faster the reaction takes place and in addition of the concentration of the catalyst has a great effect on the yield (Muhammad, et.al 2014).

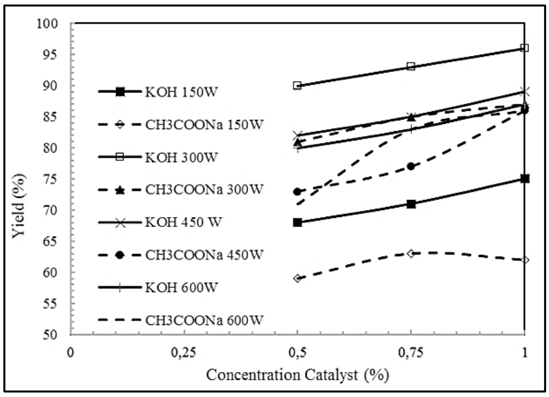

Fig. 3. Effect of catalyst concentration on yield of biodiesel

Figure 4 show that the use of CH_3COONa compared to KOH. The best yield can be obtained at power of 300 W power and catalyst concentration of 1% (w/w) of oil), reduced yield to 9% (from 96% to 87%). Fromm the above data it can be concluded that the performance of KOH catalyst is better than CH_3COONa catalyst.(Avhad and Marchetti, 2015).

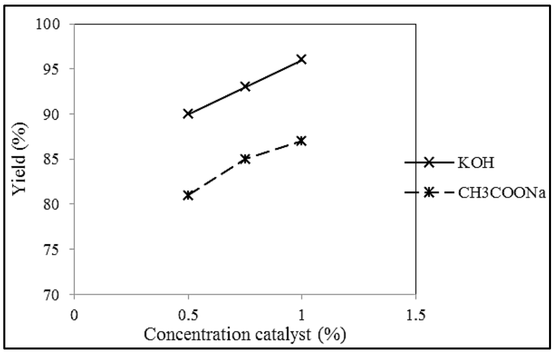

Fig. 4. Effect of base lewis catalyst on biodiesel yield on 300 W microwave power

Table 3 shows the quality characteristics of biodiesel or FAME of *Calophyllum* *inophyllum* oil at catalyst concentration 1% (w/w) oil and at power of 300 W power. From the **Table 3** can be seen that kinematic viscosity, and density meet the SNI parameters. It indicate catalytic concetration give effect on biodiesel quality (Avhad and Marchetti, 2015).

Table 3. Quality of biodiesel (FAME)

Test	SNI 04-7182-2006	KOH	CH_3COONa
Density at 40°C (gr/cm^3)	0.85 – 0.89	0.8676	0.8713
Kinematic Viscosity at 40°C (cSt)	2.3 – 6.0	3.7724	4.7678

CONCLUSION

The conclusion that microwave radiation with base lewis catalyst can be used in the process of making biodiesel from *Calophyllum inophyllum* oil. The best operating conditions at microwave power of 300 W, catalyst content of 1% CH_3COONa catalyst). When viewed from the SNI, the kinematic viscosity of the product is 3.7724 cSt (for KOH catalyst) and 4.7678 cSt (for CH_3COONa catalyst). All Catalyst has been meet standard for both viscosity and density. This suggests that *Calophyllum inophyllum* oil is one of the potential resources when used as a biodiesel feedstock considering the abundance.

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