SYNTHESIS OF BIODIESEL FROM CALLOPHYLLUM INOPHYLLUM OIL USING BASE CATALYST Na_2SiO_3/ Fe_3O_4

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1. Introduction

The fuel Oil needs in Indonesia has been increasing along with the development of technology, while the Petroleum energy reserves are reduced every day and can not be renewed. To anticipate the need for alternative energy that can be updated. The Government has issued INPRES no. 1 of 2006 on the supply and utilization of biofuels as a fuel source. The policy has established renewable resources such as biodiesel as a target for energy development (Qiqmana and Sutjahjo, 2014). So fuel-based vegetable oil is expected to reduce dependence on non-renewable fuels.

One of the vegetable oils that are most used as biodiesel feedstock is palm oil, but it will compete with the needs of human food. Vegetable oil sources that don't compete with the needs of human food such as rubber seeds, jatropha seeds, and *Calophyllum inophyllum* (CI) seeds. However, the productivity of rubber seeds is 2 ton/ ha/year and jatropha seeds is 5 ton/ha/year (Bustomi dkk, 2008).

The CI seeds has the highest productivity compare to other materials, which is 20 ton/ha/year. The oil content of CI seeds is 40-73%, while the rubber seed is 40-50 % and the jatropha seeds is 40-60 % (Soerawidjaja, 2006). The area of CI plant currently reaches 255,350 in Sumatera Barat, Riau, Jambi, Sumatera Selatan, Lampung, Jawa, Kalimantan Barat, Kalimantan Tengah, Sulawesi, Maluku dan NTT. Thus, CI oil is a renewable energy without having to compete with food needs and potential as a basic material of biodiesel (Bustomi dkk, 2008).

Biodiesel is the one of renewable energy. It is a name of alkyl ester derived from vegetable oils and animal fats. The main components of vegetable oils and animal fats are triglycerides (Qiqmana dan Sutjahjo, 2014). The advantages of biodiesel are renewable energy, has a high cetane value, low volatility, sulfur free, environmental friendly, non-toxic and biodegradable (Machmud, 2009).

During the process of making biodiesel usually was used NaOH or KOH as homogeneous catalyst which has the disadvantage the formation of side products such as soaps and complexity of the separation of catalyst (Padil dkk, 2010). To solved these problems, it's necessary to conduct biodiesel research using heterogeneous catalyst (solid). Based on Subagjo (2012), heterogeneous catalysts have several advantages, which are high catalyst activity, relatively low cost, not corrosive, environmental friendly, and can be separated from the production solution so that it can be reused.

2. Methode

The materials in used is CI oil from Koperasi Jarak Lestari Cilacap Jawa Tengah. Palm oil fly ash, iron powder, and NaOH for catalyst process. The other chemical material are aquadest, phospat acid, metanol, etanol, $H_2SO_{4,}$ KOH, dan indicator PP.

The tools in used are strainer 100 mesh, a set of mixing motors, ovens, furnaces, analytical scales, three-neck flask reactors, condensers, hot plates, thermometers and glassware such as beaker, separating funnel, and so on.

Na_2SiO_3/Fe_3O_4 Catalyst Process

Fly ash in the furnace to remove carbon and other minerals at a temperature 700 °C for 3 hours.

Then SiO_2 found in fly ash is extracted using NaOH which will produced Na_2SiO_3. The next stage, iron powder added to the extract of fly ash has been obtained from the previous stage. Then do the precipitation process at a speed 300 rpm and CO_2 streaming into that for 2 hours to produced the catalyst precipitated. After that, separated between solid and filtrate. The catalyst solid dried in the oven at 105°C. The catalyst is crushed and sieved at 30-60 mesh. Then calcined using furnace at 300°C.

Biodiesel Process

The degumming process is a process to remove impurities in the oil. The oil is weighed and then heated at temperature 80°C using magnetic stirrer. After that phospat acid added as much as 0.3% of weight of oil. The oil temperature is maintained for 15 minutes while stirring. Furthermore, the oil is inserted into the separating funnel to be separated between oil and impurities (Sahirman, 2009). The oil analyzed density, viscosity, water content, and fatty acid levels.

Esterification Process

CI oil have high free fatty acid content so it needs to be done esterification process first (Sahirman, 2009). Put 100 grams weight oil and fed into the reactor. The process is using a heater at temperature 60 °C. After that, added methanol with a ratio mol oil:methanol 1:12, H_2SO_4 18 M, and catalyst 1%-v of oil. After 3 hours, the esterification product will proceed to the transesterification process (Atabani and Cesar, 2014).

Transesterification Process

The oil is heated at 60 ℃. After the temperature is reached, methanol is added with a ratio mol methanol:oil 6:1 and Na_2SiO_3/Fe_3O_4 Na2SiO3 / Fe3O4 1%-w oil added to the reactor. After the process for 120 minutes, the oil filtered with whatman filter paper. The precipitate of the catalyst is separated from the filtrate. The filtrate is continued to the process of separation and purification of water biodiesel (Setiadi et al, 2015).

Separation adn Purification Process

The result of transesterification process is separated from the catalyst by using magnet. Then the filtrat fed into the separating funnel and have two layers. The bottom layer of glycerol is separated from the top layer of crude biodiesel. The crude biodiesel is washed by using aquadest heated at 60℃. Then biodiesel is heated using oven at 105℃. Furthermore, biodiesel is weighed to determine the yield of biodiesel produced (Ho et al, 2014):

$$Yield\ (\%) = \frac{\text{Mass of biodiesel}}{\text{Massa of oil}}\ x\ 100 \quad ....(1)$$

3. Result and Discussion

First CI oil is done with degumming process which aims to remove gum, phospholipid, and other impurities (Sahirman, 2009). The CI oil nyamplung is analyzed characteristics, which are density, viscosity, water content, and free fatty acid content. Characteristics of oil nyamplung can be seen in Table 3.1.

Table 3.1 CIO Characterization

Characterization	**Satuan**	**Before *degumming***	**After *degumming***
Density	kg/m^3	948	922
Viscosity	cSt	6,7	6,25
Water Content	%	7,01	6,88
FFA Content	%	23,5	21,69
Color	-	Green	Red chocolate

In Table 3.1, it’s known CI oil still has high water content and free fatty acid content which is equal to 6,88% and 21,69%. The high value of water content in the oil will cause hydrolysis, and high value of free fatty acid will cause a soap reaction so that reaction result low yield biodiesel (Omar and Amine, 2011).

Therefore, CI oil should be done with esterification process because of the high levels of FFA and water content will affect the quality of biodiesel.

The esterification process aims to lower free fatty acid levels. The free fatty acid levels for transesterification reactions allowed is <2% (Sahirman, 2009). After the esterification process, the FFA of CI oil decreased from 21.69% to 1.89% and the water content of CI oil decreased from 6.88% to 0.43%.

Biodiesel Characterization

The physical properties Characterization analysis of biodiesel is being compare to Indonesian biodiesel quality standards so that it can be used. The quality standard used is SNI 7182: 2015.

Table 3.3 Characterization Biodiesel Result

Parameter	SNI 7182:2015	Biodiesel Result
Density (kg/m^3)	850-890	876,18
Viscosity (cSt)	2,3-6,0	4,63
Acid value (mg-KOH/g)	Maks. 0,5	0,49
Flash point (ºC)	Min 100	107

The characteristics of biodiesel include density (ASTM D 1298), kinematic viscosity (ASTM D445), acid value (AOCS Cd 3-63) and flash point (ASTM D 93). The comparison of biodiesel characterization results of this study with SNI 7182: 2015 can be seen in Table 3.3.

4. Conclusion

Based on the research result, it can be concluded that the biodiesel produced has fulfilled the quality standard according to SNI 7182: 2015.

5. References

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