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Characteristics of *Kemiri Sunan* (*reutalis trisperma* (blanco) airy shaw) biodiesel processed by a one stage transesterification process

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Abstract. *Kemiri sunan* (*Reutalis trisperma* (blanco) airy shaw) is a plant that has the potential to be developed as a biodiesel feedstock in Indonesia because its fruit seeds have several advantages compared to jatropha and palm oil, including its oil content and productivity. The biodiesel production process consists of three stages, namely, esterification, transesterification, and purification. The process requires strong mixing to obtain sufficient contact between the vegetable oil and the catalyst or alcohol, especially at the beginning of the reaction. It is carried out manually in the reaction process through mechanical mixing (stirring) and heating in the transesterification process. Furthermore, biodiesel production process is greatly influenced by the accuracy of concentration and type of catalyst. This study aims to identify the optimal method for the process of biodiesel production from *kemiri sunan* oil. The research was conducted with a one-stage transesterification to determine the biodiesel yield and quality. The results showed that several biodiesel parameters satisfied the Indonesian National Standard (*SNI*) for biodiesel (*SNI* number 7182:2015). However, the kinematic viscosity of biodiesel was still higher than the standard.

1. Introduction

Kemiri sunan (*Reutealis trisperma* (blanco) Airy Shaw) is a plant originating from the Philippines that can grow and adapt well in various environmental conditions in many regions in Indonesia. This plant can grow well in the lowlands to 1,000 m above sea level, as found in West Java. It grows as a stand of trees, having a height of up to 75 m, also a wide and dense canopy. Furthermore, the plant is able to withstand raindrops falling directly to the ground, thereby reducing erosion and increasing water absorption into the soil. This plant also has a lot of root systems and deep taproots to prevent landslides. Thus, it is very favorable to be used as land conservation plants.

Kemiri sunan produces fruit with seeds containing high vegetable oil. Its productivity per hectare is comparable with other biodiesel-producing plants, even with palm oil, which is currently the largest oil producer [1]. *Kemiri sunan* oil is a toxic substance, with a composition of 10% palmitic acid, 9% stearic acid, 12% oleic acid, 19% linoleic acid, and 51% a-elaeostearic acid [2]. The a-elaeostearic acid content explains toxins in *kemiri sunan* oil. Therefore, it cannot be used as food. Consequently, there will be no competition between the use of biodiesel feedstock and the use of food. *Kemiri sunan* can also be used for various other purposes, including natural insecticide that is highly effective for killing pests and coating materials for ship paint [3].

Several studies on biodiesel production from *kemiri sunan* have been carried out. Djenar and Lintang (2012) researched the process of esterification of *kemiri sunan* oil *(Aleurites trisperma*) [4]. Anggraini et al. (2013) studied biodiesel's synthesis and characterization from *Reutealis trisperma* oil with the KOH catalyst [5]. Aunillah and Pranowo (2012) examined *kemiri sunan* biodiesel characteristics using a two-stage transesterification process [6]. Holilah et al. (2014) examined the effect of the amount of catalyst on biodiesel production from *Kemiri sunan* [7].

Biodiesel production generally uses homogeneous base catalysts such as NaOH and KOH because they are considered to have a higher catalyst ability than other catalysts. However, these catalysts have the disadvantage of being difficult to separate from the reaction mixture; therefore, it cannot be reused and will eventually be disposed of as waste that can pollute the environment [8]. The use of homogeneous catalysts in biodiesel makes the refining process difficult [4,5]. Likewise, biodiesel yields produced do not meet ASTM standards on several parameters [6,7,9]. Using a homogeneous catalyst is not profitable because of the formation of a by-product in soap and the difficulty of separating the biodiesel product from the catalyst [8]. Given these weaknesses, a reasonable alternative in making biodiesel is to use heterogeneous catalysts. Transesterification is the reaction between oil or fat with alcohol to form esters (biodiesel) and glycerol using a catalyst base, NaOH or KOH, to accelerate the reaction and improve the final result. It is an equilibrium reaction. In order to encourage the reaction to move to the right, it is the necessity to use an excessive amount of alcohol [10]

Aunillah and Pranowo (2012) researched producing *kemiri sunan* biodiesel using a two-stage transesterification method. With this method, the yield of *kemiri sunan* biodiesel from crude oil is 88% [6]. Whereas Holilah et al. (2013) studied that the production of biodiesel from *kemiri sunan* oil with two reaction stages, namely esterification with H2SO4 catalyst and transesterification using NaOH catalyst. The results showed that biodiesel yield increased with increasing catalyst concentration from 0.5–1.0 wt%, yet with increasing catalyst concentration from 1.5–2.0 wt%, the yield decreased. The optimum yield of 84.7% was achieved, with a catalyst concentration of 1 wt% under reaction conditions of 65°C temperature, 1-hour reaction time and 1:2 methanol/oil ratio (wt/wt) [7].

Meka et al. (2007) examined the effect of NaOH catalyst concentrations on reaction times at temperatures of 50°C and 60°C using sunflower oil as raw material. The methanol/oil molar ratio is maintained at 6:1. The study concluded that the reaction time decreased proportionally with increasing catalyst concentration from 1 to 2%. However, soap is formed when the catalyst concentration is more than 2% [11]. Rashid and Anwar (2008) found that biodiesel production from rapeseed increased at KOH catalyst concentration between 0.25–1.5%, with an optimal value at 1% [12]. Pranowo (2009) examined the process of biodiesel production in *Kemiri sunan* through the transesterification process. Of the nine criteria observed, three of them did not meet *SNI* criteria, namely kinematic viscosity, total glycerol, and alkyl ester content [13].

This study's objective is the process of transesterification, explicitly studying the effect of the percentage of base catalyst (NaOH) and reaction temperature on several parameters of biodiesel quality and yield of *Kemiri sunan* biodiesel and comparing it with *SNI* (*SNI*-04-7182 -2006).

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Table 1. Standard of chemical and physical characteristics of biodiesel.

Parameter	Unit	Indonesia *SNI* 047182	Europe EN 14214	USA ASTM D6751
Specific gravity @40°C	kg/m^3	850–890	860–900	860–900
Kinematic viscosity @40°C	cSt	2.3–6.0	3.5 –5	1.9 –6
Cetane number	-	Min. 51	51	47
Flashpoint	°C	Min. 100	Min. 101	130
Cloud point	°C	Max. 8	-	3 -12
Phosphor	mg/kg	Max.10	10	Max. 1
Copper strip corrosion		Max. no. 3	Class 1	Max. no. 3
Carbon residue	% vol	Max. 0.05	Max 0.03	Max. 0.05
Water and sediment	% vol	Max. 0.05	Max. 0.05	Max. 0.05
Distillation temperature @90% dist Vol.	°C	Max. 360		Max. 360
Sulfated ash	% mass	Max. 0.02	Max. 0.02	Max. 0.02
Sulphur	mg/kg	Max. 100	Max. 10	Max. 50
Acid number	mg KOH/g	Max. 0.8	0.5	Max 0.5
Free glycerol	% mass	Max. 0.02	0.02	Max. 0.02
Total glycerol	% mass	Max. 0.14	0.15	Max 0.14
Esther Alkyl content	% mass	Min. 96.5	Min. 96.5	-
Iodium number	% mass	Max. 116	120	-

Source: *Badan Standarisasi Nasional* (2015), European Commission (2007)

2. Methodology

Kemiri sunan biodiesel research was conducted at the Mechanical Engineering Laboratory of PGRI University Semarang. The biodiesel reactors were equipped with condensers, magnetic stirrers of variable speed, thermostats with variable temperatures, and timers with variable time. The reactor, which had been equipped with a control function on several variables, made it easy to control research variables, including reaction temperature, rotation speed, processing time. *Kemiri sunan* biodiesel was obtained from crude oil of *kemiri sunan*, resulting from oil extraction from its seeds. By using an expeller machine, *kemiri sunan* seeds were pressed then the crude oil was collected. The characteristics of *kemiri sunan* crude oil can be seen in table 2.

Table 2. Chemical and physical characteristics of *kemiri sunan* crude oil.

No	Parameter	*Kemiri sunan* Crude Oil	Unit	Method
1	Acid number	13.57	mg KOH/g	volumetric
2	Free fatty acid	0.21	%	volumetric
3	Water content	trace	% Vol	ASTM D95
4	Specific gravity	0.9273	-	ASTM 1298
5	Density at 15°C	0.9273	gr/ml	Calculated
6	Kinematic Viscosity at 40°C	64.73	cSt	ASTM D 445
7	Color	Yellow		

The transesterification process of *kemiri sunan* crude oil was carried out by reacting *kemiri sunan* crude oil with methanol and NaOH catalyst, using oil to methanol molar ratio of 1:6, catalyst amount of 0.5% NaOH weight to oil weight, at 60°C with the reaction time of 90 minutes and stirring speed of 325 rpm. The biodiesel product obtained was separated from the glycerol layer. Separated biodiesel was then washed with distilled water and analyzed for its physical and chemical properties. Parameters analyzed were the following standard biodiesel parameters in *SNI*, including specific gravity (ASTM D 1298), kinematic viscosity at 40°C (ASTM D 445), cetane number (ASTM D 613), acid number (FBI-A01-03), density, saponification value, and heat value.

Biodiesel yield is obtained by comparing the final volume of biodiesel with the volume of oil used in the reaction process:

$$\text{Biodiesel yield} = \frac{biodiesel\ volume}{crude\ oil\ volume}$$

Figure 1. Biodiesel reactor.

3. Results and discussion

The transesterification process was able to convert crude *kemiri sunan* oil into diesel fuel (table 3). It aims to transform tri-, di-, and monoglycerides with high molecular weight and viscosity that dominate vegetable oils' composition into fatty acid methyl esters (FAME), which have lower molecular and viscosity levels. The results demonstrated a large decrease in kinematic viscosity from crude oil (64.73 cSt) to be only about 10% in the biodiesel product (6.93 cSt). However, this number is still above the biodiesel standard, according to *SNI* 047182, with a maximum number of 6 cSt. Kinematic viscosity plays a very important role in the fuel spray and combustion process. Due to biodiesel blends' high viscosity, fuel tends to form larger droplets, which can cause poor fuel atomization, increases in engine deposits and wear of fuel pump elements and injectors, causing incomplete combustion processes and engine loss of power [16].

Density and viscosity are essential properties of biodiesel, which affect engine performance. The analysis results showed that the transesterification process was also able to reduce the density of *kemiri sunan* oil from 927.3 kg/m^3 to 878 kg/m^3. This showed that the ability of the flow rate of *kemiri sunan* oil increased after the transesterification process. A diesel engine's performance characteristics are influenced by density, which affects the mass of fuel injected into the combustion chamber and thus the air-fuel ratio. A change of biodiesel blend density will influence the engine's output power due to a different mass of fuel injected [16].

Table 3. Physical and chemical properties of *kemiri sunan* biodiesel.

Parameter	Unit	*Kemiri sunan* Biodiesel	Indonesia *SNI* 047182	Europe EN 14214	USA ASTM D6751
Density	kg/m^3	878	NA	NA	NA
Specific gravity	kg/m^3	878	850–890	860–900	860–900
Kinematic viscosity	cSt	6.939	2.3–6.0	3.5–5	K1.9–6
Acid number	mg KOH/g	0.496	Max. 0.8	0.5	Max 0.5

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Parameter	Unit	*Kemiri sunan* Biodiesel	Indonesia *SNI* 047182	Europe EN 14214	USA ASTM D6751
Saponification number	mg KOH/g	201.886	NA	NA	NA
Caloric value	MJ/kg	42.04	NA	NA	NA
Cetane number	-	52.2	Min. 51	51	47
Yield	%	87.96	NA	NA	NA

Besides, the biodiesel production processes carried out also lead to a decrease in acid number from 13.57 mg KOH/g to 0.496 mg KOH/g. An acid number indicates the presence of free fatty acids in biodiesel. High free fatty acids can cause corrosion and soot or crust in the engine injector. The iodine number is used as a parameter for oil unsaturation or double bonds in biodiesel. Furthermore, iodine numbers indicate unsaturated molecules' tendency to react with oxygen in the atmosphere and turn into peroxide [17].

The transesterification process in vegetable oil does not affect the composition of fatty acids of raw materials used for biodiesel. This composition has a significant effect on biodiesel parameters, including density, viscosity, cetane number, and fog point [18]. The four parameters are included in the *SNI* criteria and the USA standard.

The heating value will increase with the length of the fatty acid chain. The caloric biodiesel value of *kemiri sunan* (42.04 MJ/kg) was similar to Jatropha biodiesel, but both are lower when compared to diesel (43 MJ/kg) and gasoline (48 MJ/kg). This is because biodiesel's oxygen content decreases the heating value, but the presence of oxygen is useful to perfect the fuel combustion process [18].

Cetane numbers can affect engine performance, including combustion, stability, ease of use, smoke color, sound, CO and HC emissions. This number will increase along with an increase in the length of fatty acid carbon chain and degree of saturation. The cetane number of *kemiri sunan* was 52.2, which was higher than jatropha biodiesel, was caused by its higher percentage of long-chain hydrocarbon bonds (C18) compared to jatropha oil [6]. A higher number was also obtained due to the degree of unsaturation of *kemiri sunan* oil, which is higher than Jatropha oil. A study showed that the composition of fatty acids affects biodiesel's quality, precisely the low cetane number, which correlates with a high degree of oil unsaturation [20].

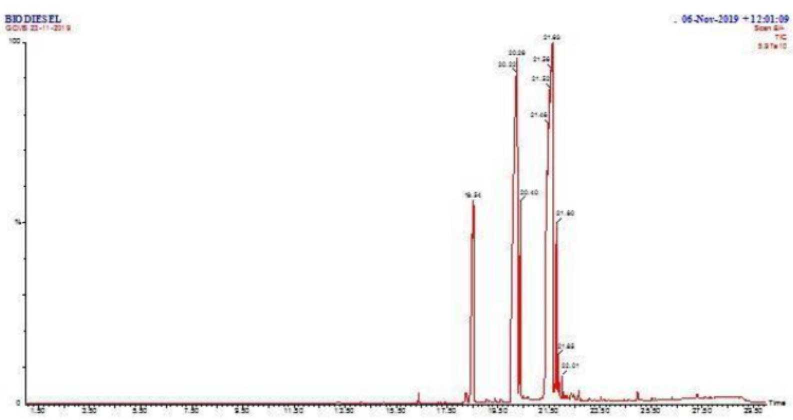

Figure 2. Biodiesel 0.3 N chromatogram.

Table 4. GC MS analysis.

No	RT	Height	Area	% Area	Content
1	18.541	3.34E+10	3.452E+09	11.97%	Palmitic acid, methyl ester
2	20.256	5.53E+10	1.014E+10	35.16%	Methyl 11-octadecenoate
3	20.401	3.21E+10	1.809E+09	6.27%	Stearic acid, methyl ester
4	21.627	5.77E+10	1.206E+10	41.83%	Methyl 9.cis.,11.trans.t,13.trans.-octadecatrienoate
5	21.802	2.84E+10	1.373E+09	4.76%	Methyl 9.cis.,11.trans.t,13.trans.-octadecatrienoate

The resultant biodiesel was characterized using Gas Chromatography (GC) to determine the methylester formed by the type of flame ionization detector. The capillary column used was a non-polar DB type with a column length of 30 m, a column diameter of 250 um, speed of flow of 100 ml/mm, flow rate of 100 ml/mm of helium and injection temperature of 250°C.

The chromatogram of the GC-MS test results shown in figure 2 described the composition of *kemiri sunan* oil. The retention time (time of separation of the tested compounds) of 18.54 was palmitic acid and methyl ester (C_17 H_34 O_2) with a percentage of 11.97%; 20.25 was methyl 11-octadecanoate (C_19 H_36 O_2) with a percentage of 35.16%; 20.40 was stearic acid, methyl ester (C_19 H_38 O_2) with a percentage of 6.27%; retention time of 21.62 was methyl9.cis., 11trans.t, 13.trans.-octadecatrienoate (C_19 H_32 O_2) with percentage of 41.83%; 21.80 was methyl9.cis., 11 trans.t, 13.trans.-octadecatrienoate (C_19 H_32 O_2) with a percentage of 4.76%.

The GC-MS results of biodiesel compounds are depicted in table 4. It showed that palmitic acid was still formed. The form of saturated fatty acids can be shown in retention time, area and constituent components. As shown in table 4, methyl ester was found in each number.; hence biodiesel was formed (methyl ester).

4. Conclusion

This study analyzed the producing performance of transesterification biodiesel from *kemiri sunan* oil that showed *kemiri sunan* biodiesel production using a 1-stage transesterification method led to biodiesel (methyl ester), producing an 87.96% yield of biodiesel from the initial oil volume. From the experimental parameters, kinematic viscosity was the only parameter not meeting the *SNI* criteria. Biodiesel compound was formed as the methyl ester.

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