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Biodiesel Production from Kapok (*Ceiba pentandra*) Seed Oil using Naturally Alkaline Catalyst as an Effort of Green Energy and Technology

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ABSTRACT: Nowadays, energy that used to serve all the needs of community, mainly generated from fossil (conventional energy). Terrace in energy consumption is not balanced with adequate fossil fuel reserves and will be totally depleted in the near future. Indonesian Government through a Presidential Decree No. 5 year 2006 mandates an increased capacity in renewable energy production from 5 percent to 15 percent in 2025. *C. pentandra seed oil* has feasibility as a sustainable biodiesel feedstock in Indonesia. The aim of this paper was to investigate biodiesel production from C*eiba petandra* seed oil using naturally potassium hydroxide catalyst. Research designs are based on factorial design with 2 levels and 3 independent variables (temperature, reaction time and molar ratio of methanol to oil). According to data calculation, the most influential single variable is molar ratio of methanol to oil. Characterization of biodiesel products fulfill all the qualifications standardized by SNI 04-7182-2006.

Keywords: biodiesel, kapok seed oil, *c. pentandra*, green technology

1. Introduction

Nowadays, energy has been gaining great importance worldwide for society to enhance economic growth and meet the standards of living. Energy that used to serve all the needs of community, mainly generated from fossil (conventional energy). Currently, 80% of conventional energy is used to general public and industries consumption (Hasan *et al.* 2012). The global energy consumption is expected to increase by 1.5% per year until 2030 (Mekhilefa *et al*. 2011; Hasan *et al*. 2012). Terrace in energy consumption is not balanced with adequate fossil fuel reserves and will be totally depleted in the near future. Furthermore, it has encouraged many reseracher to investigate the possibility of renewable resources as an alternative energy (Ministry of Energy and Mineral Resources 2011; Atabani *et al*. 2012).

Indonesian Government through a Presidential Decree No. 5 year 2006 mandates an increased capacity in renewable energy production from 7 percent to 15 percent in 2025 (U.S. Department of Commerce International Trade Administration, 2011). When the energy crisis has become a major topic in all countries, biodiesel is emerging as a very promising alternative renewable energy (Ong *et al.* 2013). Biodiesel, referred to as mono-alkyl ester of long chain fatty acids derived from vegetable oils, animal oils, alcohol, or waste cooking oil (Atadashi *etal*. 2010; Ong *et al*. 2013; Silitonga *et al*. 2013). Biodiesel is more environmentally friendly compared to conventional petroleum diesel because of its ability to reduces air pollution like carbo monoxides, hydrocarbons and particulte matter (Ong *et*

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al. 2013; Ong *et al*. 2013). Furthermore, biodiesel is a clean fuel, biodegradable, higher flash point, improved cetane number, and it can be used without any mdifications of engine (Silitonga *et al*. 2013; Ong *et al*. 2013; Ong *et al*. 2013)

Currently, more than 95% of biodiesel is made from edible vegetable oil, such as rapeseed oil (Shin *et al*. 2012), palm oil (Mekhilef *et al*. 2011), soybean oil and sunflower oil (Gui *et al*. 2008). Edible vegetable oil is considered as the first generation of biodiesel feedstock because they were the first crops to be utilized to biodiesel production (Atabani *et al*. 2012). Transesterification of edible vegetable oil into automotive fuel will cause an imbalance ecosystems, food supply, and market demand (Gui *et al*. 2008). Based on these facts, many researchers turned his attention to the non-edible vegetable oil, such as jatropha oil (Deng *et al*. 2011), sea mango (Kansedo *et al*. 2013), and rubber seed (Ramadhas *et al*. 2005). Biodiesel from non edible vegetable oil have lower price than edible vegetable oil (Singh *et al*. 2010).

Biodiesel feedstock is usually chosen based on its availability in each region or country. Indonesia is a country known for palm oil, however biodiesel production from edible oil is still being debated because it feared would disrupt food defense. In the other hand, kapok (*Ceiba petandra*) oil is one of alternative biodiesel feedstock in Indonesia. This plant, which is widely distributed in tropical region, comes from *Malvaceae* family (Ong *et al.* 2013). Old trees produce several hundred pods. Each pod contains seeds surrounded by fine fibers. The fine fiber is a mixture of lignin and cellulose (Fig. 1). Generally, people use kapok fibers as stuffing material for pillows and bolsters, while the resulting seeds discarded as waste (Ong *et al.* 2013; Silitonga *et al*. 2013). The seed can produce about 40% wt oil (dry basis), a high level of non edible vegetable oil (Ong *et al.* 2013). For this reason, it can be utilized as a potential feedstock for biodiesel production. *C. pentandra* seed oil has feasibility as a sustainable biodiesel feedstock because of its simple cultivation and short harvesting time (4 – 5 months) (Ong *et al.* 2013).

There are several technologies for biodiesel production, such as direct use (Pramanik 2003), pyrolisis (thermal cracking) (Canakci *et al*. 2009), and transesterification (Borges *et al*. 2012). Transesterfication is the most popularly and common method for biodiesel production (Borges *et al*. 2012; Ong *et al.* 2013). The process lets triglyceride present in animal or vegetable oil react with an alcohol to form ester and glycerol in the presence of homogenous or heterogenous catalysts (acid or alkaline) (Atadashi *et al*. 2010; Borges *et al*. 2012). Generally, the alkaline catalyzed production taken short time for completion, while it is not reused so they must be neutralized and discarded as salt waste (Helwani *et al*. 2009; Atadashi *et al*. 2010). Furthermore, alkaline catalyst are hazardous, caustic, and hygroscopic (Helwani *et al*. 2009).

Borges *et al.* (2012) had reviewed recent developments in heterogenous catalysts for biodiesel production Ong *et al.* (2013) conducted his research to an optimization of catalyst-free biodiesel production from *Ceiba petandra* oil (Ong *et al*. 2013). To the best of author's knowledge, there is limited study on naturally alkaline catalyst for biodiesel production from *Ceiba petandra* oil. By utilizing naturally potassium hydroxide catalyst from *Ceiba petandra* peel, it is expected to support green energy programme. The aim of this paper is to investigate biodiesel production from *ceiba petandra* seed oil using naturally potassium hydroxide catalyst.

Fig. 1 Morphology of *ceiba petandra*

2. Material and Method

2.1Material

Ceiba pentandra seed oil and naturally potasium hidroxide catalyst were obtained from home industry which was located in Pati, Central Java. Methanol (96% purity) and Sulfuric acid (H_2SO_4 97% purity) were purchased from Merck.

2.2 Transesterification process

Variables used in this transesterification process refer to Table 1. Research designs, in Table 1, are based on factorial design with 2 levels and 3 independent variables. This method was used to determine which variables are the most impact in the biodiesel production.

Naturally potasium hydroxide catalyst 1% (v/v) and *Ceiba pentandra* seed oil, in accordance with the research design, were poured to a three-neck flask and stirred 400 rpm for a predetermined time. This reaction was carried out at temperature which has been designed. Upon completion of the reaction period, the mixture were left for 24 hours to remove the top layer (methyl ester – biodiesel product) and the bottom layer (methanol and glycerol – by product). Furthermore, the bottom layer was drawn off. Then the methyl ester was dried at 100°C to remove water, methanol, and glycerol completely. The last step of this point was calculating yield on each run. Schematic overview of the experimental equipment can be seen in Fig 2.

Table 1.
Reseach designs

Run	Variables			Respond
	Temperature (°C)	Times (minutes)	Ratio (methanol: oil)	Yield (%)
1	30	30	6:1	√
2	70	30	6:1	√
3	30	120	6:1	√
4	70	120	6:1	√
5	30	30	15:1	√
6	70	30	15:1	√
7	30	120	15:1	√
8	70	120	15:1	√

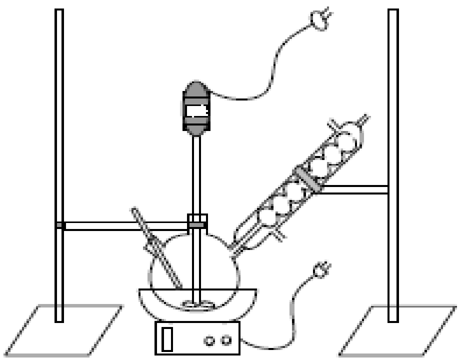

Fig. 2 Schematic overview of the experimental equipment

2.3 Analysis

Analysis of Ceiba pentandra seed oil

The free fatty acid percentage (%FFA), acid and saponification number were determined by a method based on SNI 01-3555-1998 (oil and grease analysis). Free fatty acid percentage (%FFA) is the number of gram for fatty acid in o.1 L of oil. While, acid number is the milligrams of potassium hydroxide required to neutralize free fatty acids (FFA) in 1000 milligrams of oil sample.

Analysis of biodiesel product

Characterization of biodiesel was determined based on "Standar Nasional Indonesia" SNI 04-7182-2006 (biodiesel analysis). Acid and saponification number were measured following ASTM D-664 and AOCS Cd 3-25. Cetane number was determined following ASTM D 613. Density, viscosity, and cloud point were analyzed following ASTM D 1298, ASTM D 445, and ASTM D 2500. Total glycerol consentration was measured following ASTM D-6584. While alkyl ester consentration was calculated using equation 1.

$$\frac{100(A_s - A_a - 4{,}57G_{ttl})}{A_s} \quad \textbf{(1)}$$

As : saponification number (mg KOH/g biodiesel)
Aa : acid number (mg KOH/g biodiesel)
Gttl : total glycerol consentration (%w)

3. Results

3.1 Ceiba pentandra seed oil properties

The analysis of biodiesel feedstock (*C.pentandra* seed oil) is presented in Table 2. The crude *C. pentandra* oil was found containing saponification number of 172.550 mg KOH/g biodiesel which indicated that the oil has high value of free fatty acid (FFA) (17.92%). The high value of FFA may increase the possibility of side reaction (saponification process). This process must be very avoided in the transesterification reaction.

The FFA values allowed for biodiesel production should be less than 2% (Ong *et al*. 2013). Therefore the esterification process must be conducted. Esterification is reaction between carboxylic acids and alcohols to form ester. *C. pentandra* oil, in the presence of acid catalyst (H_2SO_4 97%), was reacted with alcohol (methanol). The degree of FFA reached 1.05% after this reaction, thus fulfill the requirements to be used in transesterification process.

Tabel 2.
Initial characterization of *Ceiba pentandra* seed oil

Parameter	Value
Acid number	35.952 mg KOH/g biodiesel
Saponification number	172.550 mg KOH/g biodiesel
Free Fatty Acid (FFA)	17.97% (mg FFA/mg oil)

Acid number (35.952 mg KOH/g biodiesel), that is known from initial analysis, was used to calculate the required reagent in transesterification. Ong *et al.* (2013) reported that *C. pentandra oil* also contains a pair of unique cyclopropene fatty acids which have a higher reactivity more than double bond carbon, thus reduces oxidation stability in vegetable oil.

3.2 Effect of temperature

The reaction between *C. pentandra* oil and methanol was carried out in the difference of temperature (30° and 70°C). Research design for this variable is shown in Table 3. In general, yield values at each points has decreased. The increase of temperature (30° to 70°), causes yield values decline significantly. The greatest decrease (69.1%) occured in point 1, when the reaction were performed for 30 minutes and methanol:oil ratio (6:1). The amount of yield decline at point 2 and 4, respectively 66.34% and 55.97%. The temperature difference doesn't give a significant effect in point 3. Decrease in yield was only 0.5% at poin 3.

Table 3.
Effect of Temperature on Transesterification Reaction

Point	Variables			Yield (%)
	Temperature (°C)	Times (minutes)	Ratio (methanol:oil)	
1	30	30	6:1	87.32
	70	30	6:1	26.90
2	30	120	6:1	80.44
	70	120	6:1	27.00
3	30	30	15:1	89.10
	70	30	15:1	88.59
4	30	120	15:1	80.95
	70	120	15:1	35.64

High temperature has a negative effect on the hydrocarbon bonding thus the mixture of methanol-oil becomes more homogenous. The side reaction of esterification is greater, that there will be water accumulation in the system. This conditions would make some amounts of oil and methanol are not available for methyl ester reaction, due to partial hydrolysis of triglycerides and methanol-glycerol side reaction.

3.3 Effect of esterification times

The effect of reaction time on transesterification reaction is presented in Table 4. The reaction was conducted for 30 and 120 minutes to evaluate the effect of time reaction on yield values. According to Table 4, the yield values of each point decrease when was operated for 120 minutes than 30 minutes. Yield reduction occurred at point 5 and 8 are 7.87% and 0.15%. A significant decreases, about 59.77%, happened in point 6.

Ong *et al.* (2013) contrastly reported that the prolonged reaction time allows a longer contact between oil and methanol, thereby larger portion are converted to methyl ester. Transesterfication reaction is a reversible reaction (Fig. 2) (Helwani *et al.* 2009). In this study, the time required to achieve maximum product has been exceeded. As maximum product has been obtained and the reaction time was still running, then apply law of balance. Equilibrium law, based on Le Chatelier's principle, states that as a product concentration is greater than reactant concentration, then the reaction will shift towards the left-hand side, so that yield values will reduce.

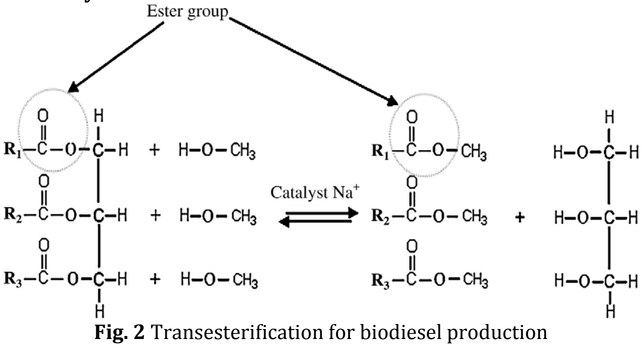

Fig. 2 Transesterification for biodiesel production (Helwani *et al.* 2009)

Table 4.
Effect of Times on Transesterification Reaction

Point	Variables			Yield (%)
	Temperature (°C)	Times (minutes)	Ratio (methanol:oil)	
5	30	30	6:1	87.32
	30	120	6:1	80.44
6	70	30	6:1	26.90
	70	120	6:1	27.00
7	30	30	15:1	89.10
	30	120	15:1	80.95
8	70	30	15:1	88.59
	70	120	15:1	35.64

3.4 Effect of methanol to oil ratio

The effect of the methanol to oil ratio on transesterification reaction was conducted within 6:1 and 15:1 methanol to oil ratio. According to Table 5, there were increments of yield values.

Stoichiometrically, methanol to oil molar ratio of 3:1 is enough to form 3 moles of methyl ester (biodiesel) and 1 mole of glycerol. However, the excess molar ratio of methanol to oil was pusposed to drive the chemical equilibrium to the right hand side (Le chatelier's principle). It ensures high conversion of triglycerides to form methyl ester.

Table 5.
Effect of Methanol to Oil Ratio on Transesterification Reaction

Point	Variables			Yield (%)
	Temperature (°C)	Times (minutes)	Ratio (methanol:oil)	
9	30	30	6:1	87.32
	30	30	15:1	89.10
10	70	30	6:1	26.90
	70	30	15:1	88.59
11	30	120	6:1	80.44
	30	120	15:1	80.95
12	70	120	6:1	27.00
	70	120	15:1	35.64

3.5 The most influential variable

Determination of the most influential variables follows the Quicker method. Code system is used to facilitate data processing (Table 6). Code of each variables is

Temperature (T) = 30 (-), 70 (+)
Time of reaction (t) = 30 (-), 120 (+)
Molar ratio of methanol to oil (R) = 6:1 (-), 15:1 (+)

Table 6.
Analysis data follows Quicker method

No	T	t	R	Tt	TR	tR	TtR	yield (%)
1	-	-	-	+	+	+	-	87.32
2	+	-	-	-	-	+	+	26.90
3	-	+	-	-	+	-	+	80.44
4	+	+	-	+	-	-	-	27.00
5	-	-	+	+	-	-	+	89.10
6	+	-	+	-	+	-	-	88.59
7	-	+	+	-	-	+	-	80.95
8	+	+	+	+	+	+	+	35.64

- Effect of T = -87.32+ 26.9- 80.44+ 27- 89.1+ 88.59- 80.95+ 35.64 = -159.68
- Effect of t = -87.32- 26.9+ 80.44+ 27- 89.1- 88.59+ 80.95+ 35.64 = -67.88
- Effect of R = -87.32- 26.9- 80.44- 27+ 89.1+ 88.59+ 80.95+ 35.64 = 72.62
- Effect of Tt = 87.32- 26.9- 80.44+ 27+ 89.1- 88.59- 80.95+ 35.64 = -37.82
- Effect of TR = 87.32- 26.9+ 80.44- 27- 89.1+ 88.59- 80.95+ 35.64 = 68.04
- Effect of tR = 87.32+ 26.9- 80.44- 27- 89.1- 88.59+ 80.95+ 35.64 = -54.32
- Effect of TtR = -87.32+ 26.9+ 80.44- 27+ 89.1- 88.59- 80.95+ 35.64 = -51.78

The influence of variable is determined by its effect. According to data calculation, the most influential single variable is methanol to oil ratio. It means that the methanol to oil molar ratio needs to be varied in subsequent studies. However, the most influential combination variables are temperature and molar ratio of methanol to oil. This combination variable should be included to the next studies.

3.6 Biodiesel Characterization

The comparison of biodiesel properties that has been resulted and biodiesel standard (SNI) is presented in Table 7. Number of SNI 04-7182-2006 was used as reference. Table 7 depicted that all parameter tested, meet the SNI standards. This achievement indicates that biodiesel produced can be tested on a motor vehicle.

Table 7.
Biodiesel characterization

Parameter	Result	SNI standard
Density (40 ^oC)	852.597 Kg/m^3	850-890 Kg/m^3
Viscosity (40 ^oC)	3.563 cSt	2.3-6.0 cSt
Cetane number	59.6	Min. 51
Cloud point	16 ^oC	Max. 18 ^oC
Acid number	0.352 mg KOH/g biodiesel	Max.0.8
Iodium number	65.27 cg iodium/g biodiesel	Max.115
Alkyl Ester	99.28 %	Min.96.5 %

4. Conclusions

C. pentandra seed oil is a very potential alternative feedstock for biodiesel production. Although the quantity of *C. pentandra* seed oil is less than palm oil, it is classified as non-edible oil, so it does not interfere food supply. According to data calculation, the most influential single variable is molar ratio of methanol to oil. Characterization of biodiesel products meet all the qualifications standardized by SNI 04-7182-2006. However, the capacity of biodiesel production should be increased and tested on a motor vehicle.

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