Effect of moisture content and amount of Hexane on in-situ transesterification of jatropha seeds for biodiesel production

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Abstract. The aim of this research is to study in-situ transesterification technique of jatropha seeds for biodiesel production. This research was conducted using a randomized factorial block design consisting of two factors that is moisture content of jatropha seeds (2% and 3%) and amount of hexane that used as a co-solvent (0 ml, 75 ml, 95 ml, 115 ml and 135 ml). The experimental result shows that the highest yield biodiesel of 78.72 % obtained at process condition of jatropha seeds moisture content of 2% and amount of hexane of 115 mL. The physico-chemical analysis of biodiesel procured the acid number of 0.35 mg KOH/g, density of 0.89 g/mL and viscosity of 3.04 mm^2/ s that meet requirement of Indonesian Nasional Standard No. 04-7182-2006.
Key words: Biodiesel, co-solvent, jatropha seeds, in-situ transesterification

Introduction

Nowadays, the price of biodiesel is higher than petroleum diesel so that its use is still limited. This is because generally the production of biodiesel using high quality raw materials that are expensive, also, the present biodiesel production technology is not yet efficient. In the future, certainly with the increasing price of petroleum diesel and along with development of biodiesel production technology that more efficient, believed the price of biodiesel will be more competitive and cheaper. Jatropha can be one of the best choice as a source of biodiesel because this plant primarily its seeds produce a high oil content used as raw material for biodiesel (Heyne, 1987), and very importantly this oil does not compete for other uses (for example, if compared with oil palm or sugarcane), as jatropha oil is not included in the category of edible oil (Hambali et al., 2006). Conventional method commonly used for producing biodiesel from jatropha oil involves various stages; oil extraction, purification (degumming, deacidification, dewaxing, dephosphorization, dehydration, etc.) and subsequently esterification and/or transesterification process. These stages spend cost over about 70% of the total production cost of biodiesel (Zeng et al., 2009).

Currently biodiesel production process has been developed using the techniques of extraction and esterification/transesterification simultaneously known as reactive-extraction process or in-situ transesterification. In in-situ transesterification process, oil extraction and esterification / transesterification performed in a single step and direct contact between the oil with alcohol. In this process alcohol has a dual role, as an extraction solvent and transesterification reagent (Georgogiannia et al., 2008). Therefore in-situ transesterification eliminates the costly hexane oil extraction step and thus reducing processing time, cost and amount of solvent required (Shuit et al., 2010).

Insolubility between oil and alcohol can obstruct the process of mass transfer and reaction rate limit. This condition can be overcome by using a stirring in the reaction system. However, this increases the cost of energy in the production process. One strategy to overcome the problem of mass transfer limitations is one phase reaction. This reaction can be formed by adding solvent to increase the solubility of the oil, the solvent here in after referred to as the co-solvent (Mahajan et al., 2006).

Beside of the amount of co-solvent, the influence of water on the kinetics of the hydrolysis reaction is very important because the high moisture content in the seeds will affect the amount of free fatty acids as well as esterification and transesterification process. The presence of excessive water can cause some reaction turned into a saponification reaction that will produce soap, the soap will react with the base catalyst and reduces catalyst efficiency thereby increasing the viscosity, gel formation and complicate the separation of glycerol with methyl ester (Fukuda, 2001; Sudradjat et al., 2005).

This research aimed to study the biodiesel production from jatropha seeds (*Jatropha curcas* Linn) using in-situ transesterification technique. The effect of moisture content of jatropha seeds and the amount of co solvent of hexane toward the quality and quantity of biodiesel obtained was investigated.

Materials and Methods

The Jatropha seeds (*Jatropha cuscas* Linn) is kindly derived from farmer in the region of Aceh Besar and Aceh Barat Daya. The hexane used was of 99 % and methanol used was of technical grade. This research was conducted using Randomized Block Design (RBD) consists of 2 (two) factors: moisture content of Jatropha seeds and the amount of co solvent of hexane.

In the beginning, fresh jatropha seeds were ground using a blender to reduce the particle size, and subsequently seeds was sieved to a size of < 1 mm. These seeds then were placed in a drying pan and were dried in a drying oven at 90^{o}C until the moisture content of seeds at 2% and 3% achieved. The dried seed was then sieved again to obtain fine particles of ≤ 0.355 mm in size. To determine maximum amount of oil that can be extracted from the seed using conventional method, a soxhlet extractor. The value of oil content of jatropha seed will be used in the calculation of the yield.

Sodium hydroxide of 0.9 g was dissolved in methanol of 400 ml, and then the mixture was placed in 1000 ml three-neck flask glass with mechanical stirrer assistance. The mixture was heated until all catalyst soluble. After desired temperature achieved (45^{o}C), 42 g jatropha seed with specified moisture content (2% and 3 %) was transferred to the three-neck flask glass and the reaction was carried out at temperature 52.5 ± 2.5^{o}C for 2 hours. In this research, in order to maximize the transesterification reaction, the amount of hexane as co solvent added at 0 ml, 75 ml, 95 ml, 115 ml and 135 ml.

The results of transesterification reaction then filtered to separate solid residue of jatropha seeds. The solid residue was washed repeatedly with methanol to recover any product that adhered to the seed and the excess methanol was removed using a rotary evaporator. After evaporation, two layers of liquid were formed. The upper layer contained the ester phase and the bottom layer contained glycerol phase. Furthermore ester phase was separated from the glycerol phase, weighed and analyzed.

Results and Discussion

Early stages of this research is the analysis of Jatropha seeds (*Jatropha curcas* L) which will be used as raw material for biodiesel. The analysis performed include analyzes of moisture content and oil content contained in the jatropha seeds. Initial moisture content of fresh jatropha seed ranges between 7.4% - 10.7% with an average value of 9.1%. In this study, the moisture content of jatropha seed is determined at 2% and 3% of wet weight, this is done to reduce to a minimum the hydrolysis reaction. High moisture content in the seeds will affect the amount of free fatty acids as well as esterification and transesterification process. High water levels can cause hydrolysis, fatty acids in biodiesel is converted into free fatty acids, thereby increasing the acid number that can corrode engine parts and injection system (Mittelbach and Remschmidt 2004).

The oil content of jatropha seed from Aceh besar is as much as 27.93% and 23.33% from Aceh Barat Daya with the average was 25.63%. The oil content obtained in this study is lower when compared to Kartika et al. (2009) which reported oil content of 38%. According to Ketaren (1986), the differences in physical and chemical properties are influenced by the type of seed plants, environmental and climatic conditions where the plants grow.

Results of physicochemical analysis showed that all parameters of biodiesel produced meets the quality requirements of Indonesian National Standard (SNI) 04-7182-2006. The analysis results can be seen in Table 1.

The yield of biodiesel produced ranged between 17.12% - 78.72% with a average of 41.62%. Figure 1 shows that treatment with 2% moisture content of jatropha seeds produces a higher yield (78.72%) compared to treatment with 3% moisture content of jatropha seeds (35.39%). Quantitatively, the lower the moisture content of Jatropha seeds are used as a feedstock for biodiesel, the higher the yield of biodiesel obtained. This is presumably due to the lower water content in the sample then chance of saponification reaction is getting smaller so that the process of conversion of oil into biodiesel to run smoothly. Fukuda (2001) and Sudradjat et al. (2005) reported that the presence of excessive water can cause some reaction turned into a saponification reaction that will produce soap, the soap will react with the base catalyst and reduces catalyst efficiency thereby increasing the viscosity, gel formation and complicate the separation of the methyl esters of glycerol. In addition to the water content, the yield of biodiesel was also influenced by the amount of hexane that used as a co-solvent.

Table 1. Comparison of the physicochemical properties of biodiesel produced in this research with the Indonesian National Standard

Parameter	Biodiesel research (average value)	Biodiesel based on SNI 04-7182-2006
Acid number (mg KOH/g)	0.35	Max 0.8
Density (g/ml)	0.89	0.86 – 0.89
Viscosity (mm^2/s)	3.04	2.3 – 6.0
Alkyl ester levels (%)	100	Min 96.5

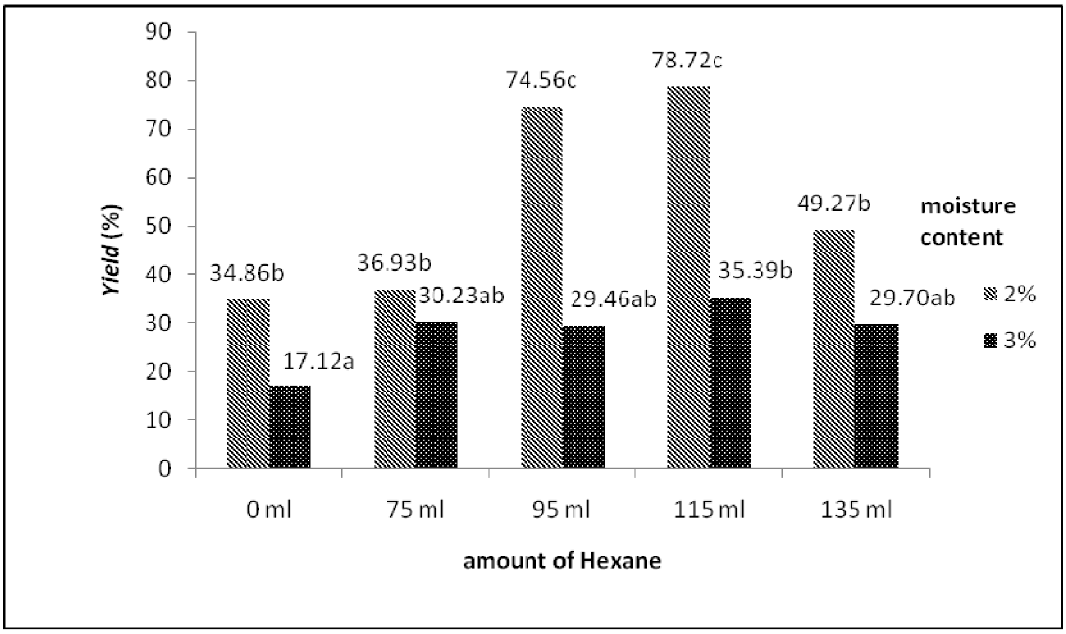

Figure 1. The interaction between treatment effect of moisture content and the amount of hexane on the yield of biodiesel from Jatropha seeds (*Jatropha curcas* L)

Figure 1 also shows that addition of co-solvent hexane as much as 95 ml and 115 ml produce yield of biodiesel higher than the addition of hexane 0 ml, 75 ml and 135 ml. However, based on the results of further tests are both have no significant differences so it can be concluded that the addition of 95 ml of hexane has reach yield optimum conditions. The amount of co-solvent required depends on the type and amount of fatty acids and triglycerides. Saturated fatty acids, unsaturated and polarity properties of this type of fat determines the amount of co-solvent that required (Boocock et al., 1996). The longer the alkyl chains on the triglyceride then level polarity become lower (become nonpolar) so that growing number of co-solvent required. Otherwise, the shorter the alkyl chain higher polarity so that the co-solvent is needed more less. This is because the amount of excess hexane resulting corelation between the hexane with alcohol toward reagent. Hexane tends binding reagent because its polarity properties, therefore, mixture process between alcohol and oil become slow so that not all the oil converted into biodiesel. The addition of co-solvent must be appropriate in order alcohols, fatty acids, triglycerides and co-solvent to form a solution of single phase (Baidawi et al., 2011).

The chemical composition of biodiesel was analyzed by a gas chromatograph coupled with a mass spectrometer detector, GC-MS (Shimadzu, Japan, Model QP 2010 Plus), using a capillary column RTX5-MS semipolar. Helium was used as carrier gas. The sample components were identified by matching their mass spectra with those from the library database. The identified main compounds in the biodiesel are Methyl Oleate (41.7%), Methyl Linoleate (30.8%), Methyl Palmitate (14.9%) and Methyl Stearate (12.6%). Methyl ester compound obtained in biodiesel products similar to fatty acids contained in the seed oil of Jatropha (*Jatropha curcas* L.). Hambali et al., (2006) states that the component fatty acids of jatropha oil is oleic acid (43.2%), linoleic acid (34.3%), palmitic acid (14.2%) and stearic acid (6.9%).

Conclusions

In-situ transesterification of *Jatropha curcas* L seed for biodiesel production was investigated in this study. Experimental results show that the moisture content of jatropha seeds and the amount of co solvent of hexane, and interaction both has very significant effect on the yield of biodiesel produced from Jatropha seeds (Jatropha curcas L). It was found that the highest biodiesel yield of 74.56% was obtained on experimental condition of moisture content of 2% and hexane amount of 95 mL. The physico-chemical analysis of biodiesel were acid number of 0.25 mg KOH/g, density of 0.89 g/mL and a viscosity value of 2.79 mm^2/s. The GC-MS analysis indicated that the amount of methyl oleate was the highest in the biodiesel.

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