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A New Route of Biodiesel Production through Chemical Interesterification of Jatropha Oil using Ethyl Acetate

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Abstract : The classical methods of biodiesel production is transesterification of triglycerides and the esterification of free fatty acid (FFA) using alcohol. Those two routes have problem in terms of by-products resulted throughout the processes. Transesterification of triglyceride and FFA esterification generate glycerol and water, respectively, as side product. Those by-products are regarded as waste in the biodiesel production. Therefore, a series of separation process is essential to achieve high purity biodiesel product. On the other hand, purification steps usually require high capital, operation, and energy cost in industrial process. Thus, to reduce separation process, a new route of biodiesel production which eliminated generation of glycerol and water by-products was proposed in this work. In this research, biodiesel production was carried out via chemical interesterification of jatropha oil with ethyl acetate over potassium hydroxide catalyst. Through this new route of biodiesel production, triacetin was yielded rather than glycerol or water. Triacetin is acknowledged as additive which functions as an anti-knocking agent for biodiesel in diesel machine. Thus, the production of biodiesel through interesterification reaction of vegetable oil with ethyl acetate would not need a further separation of by-product. In this work, the influence of main parameters on the interesterification reaction was evaluated. The effect of catalyst concentration was studied in the range of 0.5 – 1.25% w/w oil. The reaction temperature and molar ratio of oil to ethyl acetate were varied at 60 and 70°C, and 1:6, 1:9, 1:15, 1:30, and 1:60, respectively. The influence of the reaction time was tested from 1 to 8 hours. The highest reaction conversion was attained at reaction temperature of 70°C, molar ratio of oil to ethyl acetate at 1:6, 6 hours reaction time, and catalyst concentration of 0.5%.
Keywords : Biodiesel, interesterification, triacetin, jatropha oil, ethyl acetate.

Introduction

Biodiesel is a prospective renewable alternative fuel for diesel engines. It has many advantages, among others are: less polluting than petroleum diesel, contains a smaller amount of sulfur thus can extend the lifetime of the catalytic converters, high cetane number, and has caloric value which is comparable to petroleum diesel. Besides, biodiesel fuel can also be applied in conventional diesel engines without making any modification in the machine. *Biodiesel* is generally *made* of methyl esters of fatty acids, which can be produced via the two main routes, i.e. base-catalyzed transesterification reaction of triglyceride of vegetable oil or acid-catalyzed esterification of the free fatty acid (FFA)[1, 2]. The two classical routes of biodiesel production has drawback in term of the by-product resulted in the reaction. Transesterification of triglyceride with short-chain alcohols result in a large amount of glycerol as by-product, meanwhile the esterification of FFA yields water as by

product. Both glycerol and water by-products are handled as waste. Therefore, to obtain a high purity biodiesel[12-25], a series of separation processes becomes a mandatory. On the other hand, separation process is commonly one of the most energy and cost-intensive steps of a chemical process. Besides, separation process often creates a significant environmental impact due to the existence of the used separating agent, which is released as wastes[3]. Thus, reducing separation steps in chemical process industry is crucial in order to improve the efficiency and achieve the high economical process.

In biodiesel industry, separation steps can be reduced by producing biodiesel through an alternative route which avoids the formation of glycerol or water side-products. One interesting option is synthesizing biodiesel via interesterification reaction of vegetable oils by using methyl or ethyl acetate as reactant instead of short-chain alcohols. Chemical interesterification of oils with methyl/ethyl acetate will result in methyl esters (biodiesel) and triacetin, whereas glycerol would not be produced in this reaction. Triacetin is well known as fuel bio-additive which can increase fuel quality in terms of viscosity, oxidation stability, and cloud flow properties such as cloud point and pour point[4]. It also has a function as anti-knocking agent for diesel fuels.

The interesterification of oils and fats with methyl/ ethyl acetate is attractive since it can simultaneously synthesizes biodiesel and fuel bio-additive. This reaction can be written as depicted in Figure 1. Interesterification reaction is a complex reaction comprising three consecutive reversible reactions[5]. The first step is the reaction of triglyceride with ethyl acetate resulting fatty acid ethyl ester (biodiesel) and monoacetin diglyceride, the second step is the reaction of monoacetin diglyceride with ethyl acetate to result in fatty acid ethyl ester (biodiesel) and diacetin monoglyceride, and the last step is the reaction of diacetin monoglyceride with ethyl acetate yielding fatty acid ethyl ester (biodiesel) and triacetin. Hence, the entire reaction stoichiometrically requires 1 mol of triglyceride and 3 mol of ethyl acetate to produce 3 mol of fatty acid ethyl ester (biodiesel) and 1 mol triacetin. This reaction can be carried out by using various types of base or acid catalysts.

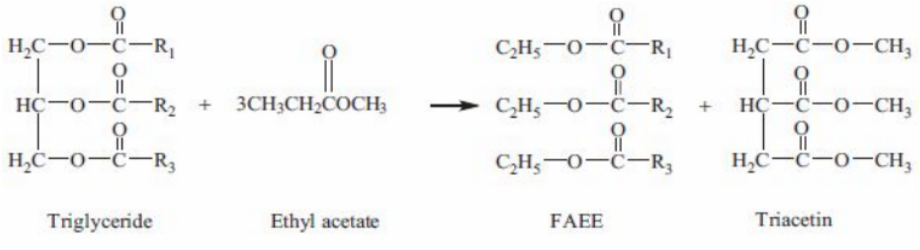

Figure 1. Interesterification of Triglyceride

Interesterification of vegetable oil is a new route of biodiesel production which could transform triglycerides into biodiesel without glycerol formation. This route is a promising way of the process intensification on biodiesel production. In this research, biodiesel production via the interesterification reaction of crude jatropha oil with ethyl acetate was investigated. Potassium hydroxide catalyst was employed to enhance the reaction rate. The experiments were conducted in a batch reactor at different temperature, reaction time, oil to ethyl acetate molar ratio, and catalyst concentration to evaluate the effects of those parameters on biodiesel yield from jatropha oil. To prevent the side-saponification reaction due to the high FFA content in oil feed-stocks, crude jatropha oil was primarily neutralized using Na_2CO_3 solution.

Experimental

A. Material

Crude jatropha oil (from PT. Pura Energi, Indonesia) and ethyl acetate (from Merck, Germany) were the main reactants for the biodiesel synthesis. Potassium hydroxide was utilized as catalysts for the chemical interesterification reaction. Physical properties of this crude jatropha oil were reported in Table I.

Tabel I. Physical Properties of Crude Jatropha Oil

Properties	Value
Density	970 kg/m3
Viscosity	38.83 mm2/s
Acid Number	17.71 mg KOH/g oil
Acidity (Acid Content)	8.9 w/w oil

B. Neutralization of Crude Jatropha Oil

Neutralization of crude jatropha oil was conducted in a 500 ml three-necks flask using Na_2CO_3. Soda ash (Na_2CO_3) was initially dissolved into aquadest to obtain saturated aqueous solution of sodium carbonate. Subsequently, 100 ml of crude jatropha oil was heated to 90°C in a three-necks flask equipped with a glass condenser. After the desired temperature was attained, a solution of Na_2CO_3 was added to the jatropha oil and stirred for 60 minutes using magnetic stirrer. During the neutralization process, saponification reaction of FFA contained in the oil with alkali took place and resulted in soap. The neutralized jatropha oil was then separated from the soap using filtration method.

C. Interesterification of Jatropha Oil

Alkaline-catalyzed interesterification of jatropha oil with ethyl acetate was carried out in a batch reactor equipped with condenser and stirrer as depicted in Figure 2.

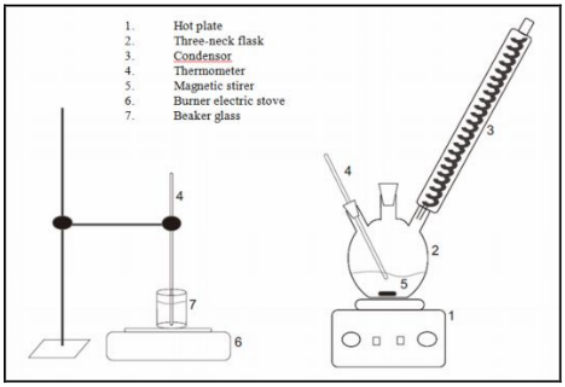

Figure 2. Batch Reactor for Interesterification Process

First of all, potassium hydroxide catalyst was dissolved into ethyl acetate in a certain concentration and subsequently poured into the reactor. The solution was then heated to the desired temperature. In the other flask, refined jatropha oil was also heated to similar temperature. After the desired temperature was reached, the oil was decanted into the reactor. Interesterification reaction of jatropha oil with ethyl acetate using potassium hydroxide catalyst was conducted in different reaction time, temperature, catalyst concentration, and molar ratio of the reactants. Catalyst concentration was varied in the range of 0.5 – 1.25% w/w oil. The effects of reaction temperature and molar ratio of oil to ethyl acetate were studied at 50, 60, and 70°C, and 1:6, 1:9, 1:15, 1:30, and 1:60, respectively. The reaction time was tested from 0 to 8 hours.

D. Analysis

Yields of biodiesel in the interesterification process was determined using *High Performance Liquid Chromatograph* (Quaternary Gradient System with UV-Vis Detector, Type LC-20 Prominence, Shimadzu) and *Gas Chromatograph - Mass Spectrometer* (Type QC-2010 Ultra El Mode, Shimadzu)

Results and Discussion

A. Neutralization Process of Crude Jatropha Oil

Neutralization is a method for chemical refining of high FFA vegetable oils. The objective of this process is to reduce FFA content in oil feedstock by saponification reaction using an alkaline solution, which results in soaps in water phase[6]. The soap produced is then separated from the oil feedstock. In this work, neutralization process was performed using Na_2CO_3. Alkaline neutralization of FFA in jatropha oil with Na_2CO_3 solution can be written in Figure 3. Carbonic acid resulted ini this reaction will dissociate to CO_2 gas dan H_2O. Carbon dioxide gas forms foam in the soap, so that the soap will float on the oil. The soap therefore can be easily separated using filtration method. In this work, rendement of the refined jatropha oil after neutralization process was 72%. The FFA content of the neutralized oil was 0.17% or it was identical to 0.3506 mg KOH/g oil. This acidity value could fulfill requirement for the reaction of oil using alkaline catalyst. Subsequent to the neutralization step, jatropha oil was used as the feedstock for the biodiesel synthesis via alkaline interesterification with ethyl acetate.

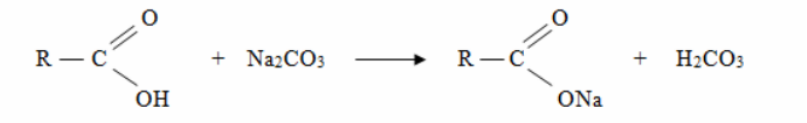

Figure 3. Neutralization Reaction using Natrium Carbonate

B. Effect of Catalyst Concentration on the Biodiesel Yield

The effect of catalyst concentration on the biodiesel yields during the alkaline-catalyzed interesterification of jatropha oil with ethyl acetate was investigated at the reaction time of 1 hours, temperature of 60 °C, and molar ratio of oil to ethyl acetate of 1:6. Potassium hydroxide catalyst was varied at 0.5, 0.75, 1, and 1.25% w/w oil. The result of the experiment is demonstrated in Figure 4.

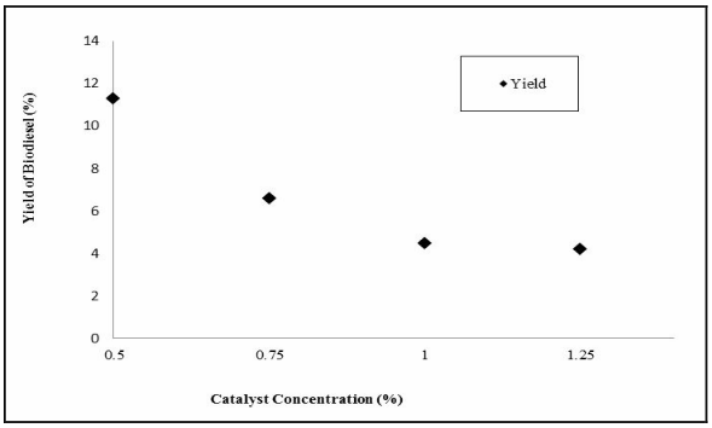

Figure 4. Effect of Catalyst Concentration on Biodiesel Yield [Reaction Condition: Temperature = 60°C; Reaction Time = 1 hr; Molar Ratio of Jatropha Oil to Ethyl Acetate = 1:6]

It was disclosed that biodiesel yield decreased with the increasing potassium hydroxide concentration. As depicted in Figure 4, an increase in catalyst concentration from 0.5 to 1.25% w/w oil brought about a corresponding reduction in the biodiesel yield from 11.27 to 4.21%. It means that catalyst concentration of 0.5% was the optimum point. Maddikeri *et al.*[5] reported the similar phenomenon in the interesterification of waste cooking oil and ethyl acetate in the presence of potassium methoxide catalyst. The decreasing yield comes about since the excessive amount of catalyst will enhance the formation of an emulsion. The emulsion leads to the formation of gels, which lowers the fraction of Fatty Acid Ethyl Ester in product mixture[7].

Chromatogram of the HPLC analysis on product mixture obtained at the reaction time of 1 hours, temperature of 60°C, molar ratio of oil to ethyl acetate of 1:6, and catalyst concentration of 0.5% is presented in Figure 5. It was found that triacetin has not been detected due to its low precentage in the product mixture. Meanwhile, ethyl acetate, methyl decanoat, and methyl oleate were found out at the retention times of 2.8, 3.277, and 4.918 min, respectively

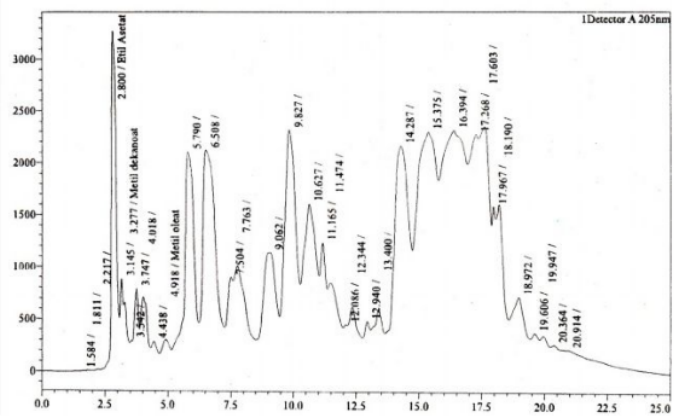

Figure 5. HPLC Chromatogram for Product Mixture [Reaction condition: Temperature = 60°C; Reaction Time = 1 hr; Molar Ratio of Oil to Ethyl Acetate = 1:6; Catalyst Concentration 0.5%]

C. Effect of Temperature and Reaction Time on Yield

The influence of operating temperature and reaction time on the yield of jatropha biodiesel in interesterification reaction was studied over the temperatures of 60 and 70°C within 0 – 8 hours reaction time. The reactant molar ratio and catalyst concentration were fixed correspondingly at 1:6 and 0.5%. The experimental result is illustrated in Figure 6.

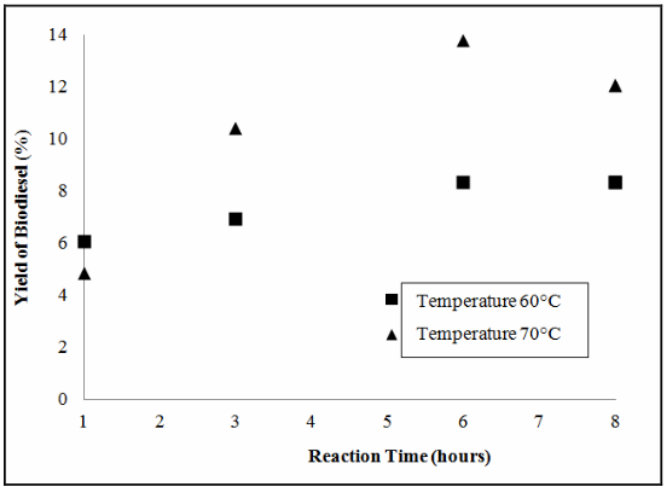

Figure 6. Effect of Temperature and Reaction Time on Biodiesel Yield [Reaction condition: Catalyst Concentration of 0.5% and Molar Ratio of Oil to Ethyl Acetate = 1:6]

It was revealed in Figure 6 that the reaction temperature of 70°C provided higher yield than that of 60°C. This finding can be described by the fact that the higher reaction temperature leads to a better solubility of ethyl acetate in the oil phase. It therefore decreases the mass transfer resistance between the two phases and

improves the contact of the reactants, resulting in the higher biodiesel yield[5]. The effect of reaction temperature on biodiesel yield can also be explained by the classical theory on kinetics and reaction rate. It is generally stated that the higher reaction temperature denotes the higher kinetic energy of the system, which causes the possibility of the higher reaction rate and conversion[2]. Furthermore, Maddikeri et al.[5] as well as Tan et al.[8] gave an account that an increase in the reaction temperature will improve the reactivity betweet ethyl acetate and triglycerides. Hence, it raises reaction rate and generates the higher yield.

The influence of reaction time is also presented in Figure 6, as well. It was demonstrated that the yield of biodiesel increased with the increase in reaction time from 0 to 6 hours. However, the yield slightly declined when the interesterification reaction was extended until 8 hours. The lessening on the yield is most likely due to the thermal decomposition of biodiesel during a prolonged reaction time of interesterification reaction[9].

D. Effect of Reactant Molar Ratio on the Biodiesel Yield

Molar ratio of reactants is a significant aspect influencing the yield of biodiesel in the interesterification reaction. Stoichiometrically, interesterification reaction requires three moles of ethyl acetate for each mole of glycerides. It indicates that the molar ratio of 1:3 is necessitated for a stoichiometric reaction. Nonetheless, interesterification reaction is a reversible reaction which usually need an excess of ethyl acetate above the stoichiometric requirement to shift the equilibrium towards the right[5]. In this work, effect of molar ratio of triglycerides in jatropha oil to ethyl acetate was evaluated at 1:6, 1:9, 1:15, 1:30, and 1:60. The other variables were maintaned constant at the reaction temperature, reaction time, and catalyst concentration of 70°C, 8 hours, and 0.5%, respectively.

The experimental data which were obtained based on the GC-MS analysis is presented in Figure 7. It was exhibited that the yield of biodiesel diminished with the increasing molar ratio of etil acetate to jatropha oil from 6:1 to 60:1. It occurred since the amount of ethyl ester (biodiesel) produced during this interesterification reaction was far less than the amount of unreacted ethyl acetate. Thus, the higher amount of ethyl acetate employed in the reaction, the smaller fraction of biodiesel in the product mixture will be found.

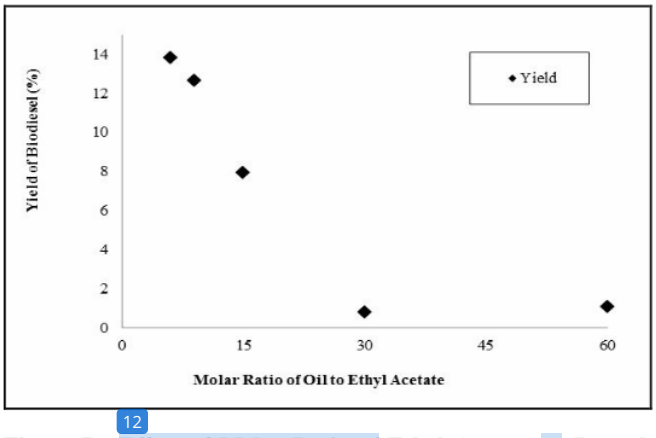

Figure 7. Effect of Molar Ratio of Ethyl Acetate to Jatropha Oil on Biodiesel Yield [Temperature = 70°C; Reaction Time = 6 hrs; Catalyst Concentration = 0.5%]

Based on the data achieved from all the experiments conducted, it was shown that the best yield was 83.79%, provided by the interesterification reaction performed at the molar ratio of jatropha oil to ethyl acetate of 1:6, catalyst concentration of 0.5%, temperature of 70°C, and reaction time of 6 hours. Biodiesel yield obtained through this interesterification can be enhanced by optimizing the process variables. The main aspect to be evaluated is the catalyst employed in the reaction. This work utilized potassium hydroxide which was dissolved in ethyl acetate reactant. Since there was no alcohol in this interesterification reaction, the active catalyst compound of potassium alkoxide cannot be form. Meanwhile, the catalytic activity of potassium oxide is significantly lower than alkoxide. Thus, for the further experiment, a small amount of alcohols (methanol/ ethanol) should be utilized to dissolve potassium hydroxide and form alkoxide which will perform as an active

catalyst for interesterification reaction. Besides, the low yield was also attributed to the existence of side reaction between potassium oxide and ester forming potassium acetate. Hence, the addition of a small amount of alcohol will convert potassium hydroxide into potassium methoxide and prevent the formation of potassium acetate[10]. It will consequently enhance biodiesel yield.

Conclusion

The application of the new route of biodiesel production through chemical interesterification of jatropha oil with ethyl acetate has been investigated in this work. Before performing the interesterification reaction, jatropha oil was neutralized using Na_2CO_3 solution to reduce the FFA content from 8,9% to 0,17%. Subsequently, the interesterification reaction was carried out in the presence of potassium oxide catalyst to enhance the reaction rate. It was demonstrated that the highest yield was obtained at the reaction temperature of 70ºC, potassium hydroxide catalyst concentration of 0.5% w/w oil, and molar ratio of oil to ethyl acetate of 1:6 with the biodiesel yield of 14%. The further research should be conducted at the higher temperature and different types of catalyst to achieve the higher reaction yield.

Acknowledgement

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