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Biodiesel Reactor Design with Glycerol Separation to Increase Biodiesel Production Yield

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Abstract

The study consisted of reactor design used for transesterification process, effect of glycerol separation on transesterification reaction, determination of biodiesel quality, and mass balance analysis. The reactor was designed by integrating circulated pump/stirrer, static mixer, and sprayer that intensify the reaction in the outer tank reactor. The objective was to reduce the use of methanol in excess and to shorten the processing time. The results showed that the reactor that applied the glycerol separation was able to compensate for the decreased use of the reactant methanol from 6:1 to 5:1 molar ratio, and changed the mass balance in the product, including: (i) the increase of biodiesel production from 42.37% to 49.34%, and (ii) the reduction of methanol in excess from 42.37% to 32.89%. The results suggested that the efficiency of biodiesel production could be increased with the glycerol separation engineering.

Abstrak

Desain Reaktor Biodiesel dengan Pemisahan Gliserol untuk Meningkatkan Hasil Produksi Biodiesel. Penelitian ini terdiri dari desain reaktor yang digunakan untuk proses transesterifikasi, pengaruh pemisahan gliserol pada reaksi transesterifikasi, penentuan kualitas biodiesel, dan analisis neraca massa. Reaktor didesain dengan mengintegrasikan pompa/pengaduk, mixer statis, dan *sprayer* yang mengintensifikasi reaksi pada reaktor tangki luar. Tujuan penelitian ini adalah untuk menurunkan jumlah methanol yang berlebih dan mempercepat waktu pemrosesan. Hasil penelitian menunjukan bahwa reaktor yang menggunakan pemisahan gliserol dapat mengkompensasi penurunan penggunaan reaktan methanol dari 6:1 hingga 5:1 rasio molar, dan mengubah neraca massa pada produk yang antara lain: (i) peningkatan produksi biodiesel dari 42,37% hingga 49,34%, dan (ii) penurunan metano berlebih dari 42,37% hingga 32,89%. Berdasarkan hasil penelitian, efisiensi produksi biodiesel dapat ditingkatkan dengan rekayasa pemisahan gliserol.

Keywords: biodiesel, design, glycerol phase separation, reactor

1. Introduction

One of the success indicators in biodiesel reactor design is the achievement of the production process conversion rate. The main parameter that determines the process efficiency is the design of control system of transesterification reaction of vegetable oil into biodiesel. The main problems in the transesterification reaction, namely are: (a) the reactants (oil and methanol) that are difficult to mix with each other because of their difference in phases, so it needs a strong and long stirring time in a batch blades-stirring reactor type, and (b) the reaction is reversible, which results in incomplete reaction. Reversible reaction is usually treated with methanol in excess at least twice as much as the theoretical requirements [1].

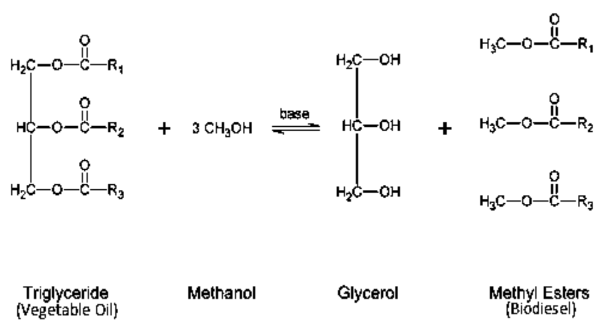

Figure 1. Transesterification Reaction

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The batch blade stirrer biodiesel reactor type has been improved by several researchers by applying a static mixer -reactor that is able to increase the reaction rate of oil and methanol in which the stirring is intensively done by the circulation pump, static mixer, and sprayer [2-3].

Application of static mixer in batch reactors only managed to speed up the conversion process the oil into biodiesel, but the use of excess methanol remains to be done to push the reaction equilibrium towards product formation corresponding to mass action law, the equilibrium reaction shifts to the party who is not given the excess mass. The addition of excess methanol as reactant will harm competition and lower the economic value of the reactor. Therefore, it is necessary to find other measures in which the transesterification reaction was restrained to the formation of biodiesel products as much as possible.

The idea of the study was how to create a reactor that is able to accommodate the separation of glycerol gradually as soon as it is formed during the process. It is an alternative to completing the reaction that is in accordance with the Mass Action Law, in which the separation of glycerol as a byproduct of the transesterification reaction is expected to compensate for the reduced use of methanol in excess and to increase the reaction rate.

The aim of this study is to design and build a biodiesel reactor for converting vegetable oils into biodiesel. In this paper, we present the results of a test on an effect of the glycerol separation on the transesterification reaction, determine the quality of biodiesel, and analyze the mass balance.

2. Methods

Reactor design. Biodiesel reactors are vessels designed to contain chemical reactions. The design of a chemical reactor deals with multiple aspects of chemical engineering. Chemical engineers design reactors to maximize net present value for the given reaction [6]. Designers ensure that the reaction proceeds with the highest efficiency towards the desired output product, producing the highest yield of product while requiring the least amount of money to purchase and operate.

The research was an engineering design research, and was not a routine research in which there were new contributions, both in processes and products [7]. The stages of the design engineering were: (i) problem identification (ii) initial idea, (iii) problem refinement, (iv) decision making, and (v) implementation. The designers generally worked in sequence according to these stages, but it was not impossible that they returned to a previous step if there were mistakes. The stages of the study were shown in Figure 2.

In order to carry out the conversion of vegetable oils into biodiesel, it is necessary to prepare a processing machine, a biodiesel reactor. However, previously, a laboratory-scale biodiesel reactor was needed with the following criteria: (i) the amount of biodiesel produced was 500 mL per working cycle (batch); (ii) the target of the design was expected to convert all types of vegetable oils into biodiesel; (iii) the materials of reactor were easy to obtain; and (iv) the design was oriented to balance the performance cost to get the maximum value.

Reactor testing. As much as 500 mL (0.527 mol, 451.4 g) of palm oil was transesterified using methanol (3.16 mol, 128 mL, 101.12 g) in the presence of 1% basic catalyst (4.5 g). The Properties of the oil were tabulated on Table 1.

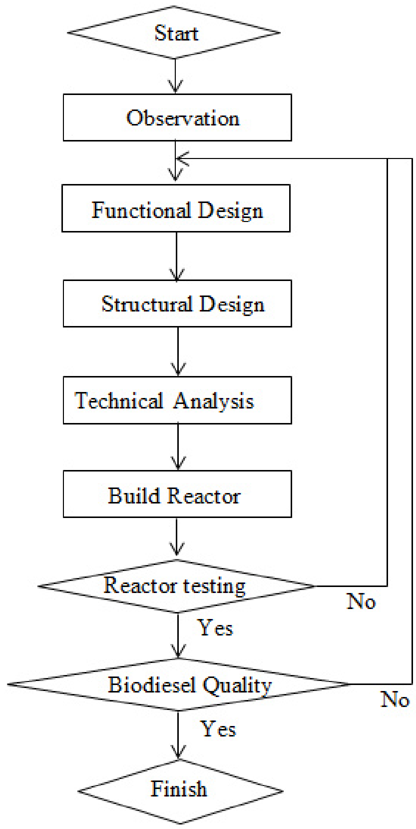

Figure 2. Flow Chart of Biodiesel Reactor Design

Table 1. Properties of Palm Oil

Parameter	Results	Unit
Relative molecular mass (M_r)	856.10	*g/mole
Density (ρ) at 40 °C	902.10	kg/m^3
Kinematic viscosity (υ) at 40 °C	47.98	mm^2/sec
Flash point	286.00	°C
Acid number	0.27	mg KOH/g

* Source: Yoeswono *et al*. [8]

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The stages of reactor testing consisted of two kinds of experiment, namely: (i) determining the reactor capacity, which consisted of the circulation flow rate, the starting time of glycerol formation and the rate of glycerol addition, and (ii) comparing the effect of glycerol treatment and checking in a pairs test design.

3. Results and Discussion

Reactor design. The functional components of the biodiesel reactors were: (i) tank reactor, (ii) circulating pump, (iii) a heating element, (iv) static mixer, and (v) sprayer. The condition in the reactor tank was much quieter because there was no pounding waves or turbulence as in the mechanism of agitator blades; liquid mixture was splashed smoothly and evenly and fell into the surface of the liquid, then gradually flew down. In a steady condition, there was available time for glycerol gradual separation from the mixture system, and it was used as much as possible to prevent the glycerol from being back-reactants. And it was also to set the position of the tank toward the exit pipe circulation pump from the bottom of the tank reactor in order to be a place of glycerol decantation.

Structural design of the reactor (Figure 3) consists of (1) glass column with diameter of 8 cm and height of 20 cm as a reaction tank; (2) circulation stirrer (Denso pump No. 06021-1480 windshield washer, voltage: 12 V; current: 3.5-3.9 A; debit: 36 cm^3/second); (3) static mixer with diameter of 8 mm equipped with spiral plat on breaking and splitting position; (4) sprayer with hole (1 mm); (5) electrical heating element SG 1103 20 Watt; (6) thermostat EGO 0913 limit 110 ^oC; (7) hand thermometer; (8) glycerol decanted zone; (9) output tap of glycerol and biodiesel; (10) distributing pipeline SMC TY 0805 polyurethane 8x5; (11) regulated DC power supply Montana SPS 7A.

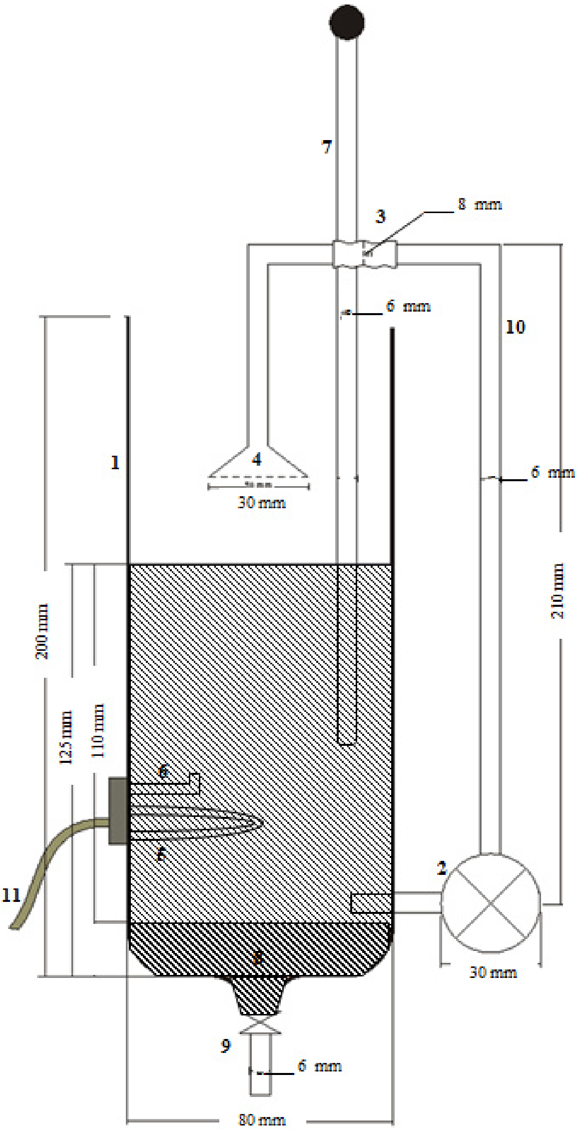

Figure 3. Biodiesel Reactor

Measurement of reactor performance: (1) Measuring the rate of stirring circulation. In a stirring circulation with a flow rate of 20.11 mL/sec, when it was processed for 20, 40, and 60 minutes, then the 620 mL mixture (500 mL oil + 120 mL methanol), respectively, had circulation of 39, 78, and 117 times evenly and effectively, because it passed through three types of mixing, namely agitation by the pump propeller, separation-reverse-rotation by a static mixer, and spraying to the surface by the nozzle. Thompson and He [6] have investigated that the use of a circulation pump and static mixer is more effective than the agitator blades. A blade stirrer has weaknesses; it consumes more energy to move the stirrer and requires a strong frame construction to support each component of the reactor. (2) Starting time of glycerol formation. The shape of this reactor has been given place of glycerol phase during the reaction. Position of inlet pipeline to the pump with 5 cm from the bottom reactor tank was to provide a quiet place for decanting glycerol along the process. This conical shape is the decantation zone of glycerol phase. The formation time of glycerol is easy to observe because the phase, color, and specific gravity was different from the ester phase. Glycerol formation is characterized by the appearance of a red-brown phase at the base of the reactor, which is in contrast with the bright yellow color of the ester phase. It was observed that the average time of glycerol formation is was 4.8 minutes after the transesterification process begun. Although this data was obtained visually, it was important information for the implementation of the following experiment. (3) Glycerol growth. Crude glycerol phase measurements carried out in stages during the process. This conical shape of the reactor tank is the glycerol decanted zone. Furthermore, glycerol phase was released gradually from valve ball at the bottom of the tank to be measured by time (Figure 4).

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The relationship between the processing time (X-axis) and an increase in glycerol volume (Y-axis) follows the inverse model described elsewhere [9]. The curve shape is a nonlinear regression model or a reciprocal model, described as follows:

$$\hat{Y} = 66.44 - 351.17\,X^{-1} \quad (1)$$

The inverse model showed that if the X-variable increased indefinitely, then the form of b_1X^{-1} would approach zero and Y would be close to the limit (asymptotic) of b_o value (intercept). The equation had the value of b_1 (regression coefficient), -351.17, and the glycerol growth would be limited to close to the intercept value 66.44.

Comparing the glycerol separation treatment and control. This experiment tried two treatments, namely: (i) P_0: control or without the separation of glycerol, and (ii) P_1: the separation of the glycerol every five minutes. Both treatments were applied in the pair test design that was repeated five times using the t-test and the outcome is presented in Table 1.

The results proved that the glycerol separation gave a different effect on the volume of glycerol, the volume of washed biodiesel, and the volume of dried biodiesel as a final product of the process. Washed biodiesel was mixed with warm water (60 ° C) and stirred for five minutes, then allowed to settle for two hours in order to obtain biodiesel and water phase separation. This work was performed with two replications. Furthermore, biodiesel is dried in the oven at a temperature of 60 ° C for one hour. The differences in biodiesel yield as end yield proved that the mechanism of separation of the glycerol by the design-build reactor gave higher yields compared with that of control.

Effect of the decrease in the level of methanol and the processing time. Rahmat *et al.* [9] reported that separation of glycerol could compensate a decrease on methanol and process. The separation of glycerol was conducted as it would be separated easily based on the density.

There were no differences in effect between the treatment of methanol m_1 through m_3 on the quantity and yield of biodiesel, it was indicated that the glycerol separation treatment could compensate the reduction of methanol in excess that should be given. In accordance with the Law of Mass Action, the transesterification reaction equilibrium position can be shifted toward the formation of biodiesel by splitting one of the products so

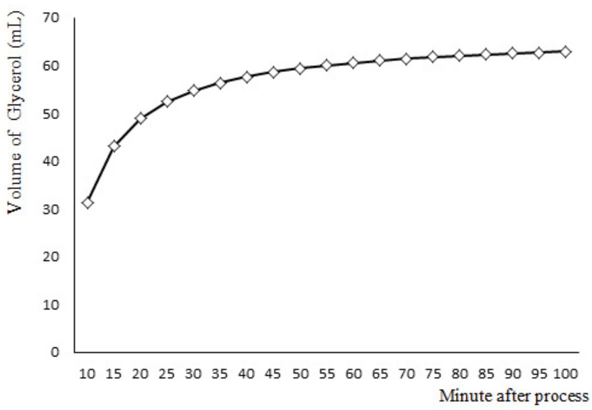

Figure 4. Glycerol Growth during the Process [9]

Tabel 1. Comparing the Effects of Glycerol Separation (P_1) and Control (P_0) on the Transesterification Products

Treatment	Volume of Product (mL)		
	Glycerol	Washed Biodiesel	Dried Biodiesel
P_0	80.00 b	496.40 a	486.60 a
P_1	75.00 a	508.80 b	499.40 b
t-calculation	2.43 *	3.01 *	2.65 *

Note: Numbers followed by the same letter indicates not significantly different according to t-Test; * = significantly different according to t-table= 2.132 (df 4; α 0.05)

Tabel 2.The Effect of Decreasing in Level of Methanol and Processing Time on the Dried Biodiesel

Treatment factors	Volume of Dried Biodiesel (mL)		Yield (%)
m_1 (ratio 6:1)	496.97	a	99.394
m_2 (ratio 5,5:1)	495.92	a	99.184
m_3 (ratio 5:1)	497.00	a	99.400
w_1 (60 minutes)	495.06	a	99.012
w_2 (50 minutes)	496.26	a	99.252
w_3 (40 minutes)	496.78	a	99.356
w_4 (30 minutes)	498.22	a	99.644
w_5 (20 minutes)	496.83	a	99.366

Note: Numbers followed by the same letter indicates not significantly different according to Duncan's Multiple range test level of 5%

Tabel 3. Quality Testing of Biodiesel Sample

Parameter tested	Unit	SNI 04-7182-2006 Biodiesel standard	Biodiesel sample	Petrodiesel*
Density (at 40 ^oC)	kg/m^3	850–890	858.39	870
Viscosity (at 40 ^oC)	mm^2/s	2.3–6.0	4.75	1.6-5.8
Cetane number	-	>51	62.00	48
Flash point	^oC	≥100	175.00	176
Acid number	mg KOH/g	<0.8	0.09	0.6
Total glycerin	%-w	<0.24	0.21	-
Methyl ester content	%-w	>96.5	98.98	-

Note: * ASTM [11]

it would not be a reverse reactant. Selection of glycerol as a by-product which was immediately separated from the reaction system has been considered more effective and practical since glycerol will separate itself by the specific gravity if it was accommodated by the shape of the reactor tank, i.e. by adjusting the position of the pipeline to the circulating pump 5 cm above the cone-shaped base. This structure would accommodate glycerol that has been formed and was not brought back into the circulation pump.

The processing time could be reduced up to 20 minutes (m_5) using the circulation pump reactor that had a static mixer and a sprayer proved more effective than a blade-stirred reactor and a continuous flow stirred reactor (CSTR). Transesterification reaction took place on condition that an effective stirring could be done to overcome the problem of mass transfer of vegetable oil into methanol.

Biodiesel quality testing. The quality testing aimed to determine the suitability of biodiesel fuel according to the requirements of diesel engines, including mainly the parameters of: (i) viscosity, as the first indications of whether the injector easily atomized the fuel in the diesel engines, (ii) density, for the completeness combustion condition of the diesel engines (iii) acid number, as an indicator of the level of free fatty acids and mineral acids, (iv) crude glycerol content, the number that shows the amount of glycerol in biodiesel after washing and drying; and (v) methyl ester content, as the indicator of the completeness of the transesterification reaction to convert vegetable oils into biodiesel [6-7]. The parameters of the test results was not merely a measure of the value of the conversion yield of vegetable oil into biodiesel, but also for quality assurance in using diesel engines to prevent it from accelerated damage. Biodiesel with high acid numbers will cause all engine fuel system components rusty. Similarly, biodiesel with viscosity, density, and total glycerol beyond the standard limit will result in incomplete combustion and will generate a lot of crust on the combustion chamber. The crust is abrasive substance when carried away by the movement mechanism of any engine components, resulting in accelerated damages.

Transesterification reaction was done to improve the characteristics of the oil feedstock. In this experiment, transesterification lowered the viscosity from 47.98 mm^2/sec to 4.75 mm^2/sec by biodiesel (Table 3). Sample used in the characterization of biodiesel was a product of transesterification with the ratio of methanol 5:1 for 20 minutes. Transesterification could optimize the properties of vegetable oil. For instance, viscosity could be reduced from 47.98 into 4.75 mm^2/s. Properties of biodiesel met the quality standard of SNI 04-7182-2006 [10] and ASTM [11].

Mass balance analysis. The transesterification reaction by applying the glycerol separation was able to compensate for the decrease of the molar ratio of reactants methanol from 6:1 (m_1) to 5:1 (m_3), which changed the mass balance in the product, including: (i) increasing the biodiesel from 42.37% (in m_1) to 49.34% (in m_3), and (ii) reducing the methanol in excess from 42.37% (in m_1) to 32.89% (in m_3). The results of this calculation proved that the efficiency of biodiesel production could be enhanced with the glycerol separation engineering.

4. Conclusions

The biodiesel built-reactor accommodated the reaction running intensely in the outer reactor tank, and at the mean time, in the reactor tank, the glycerol separation took place after being formed through the deposition mechanism.

Technology of glycerol separation on the biodiesel reactor could decrease the usage of methanol into molar ratio of 5:1 and process time of 20 minute without affecting the quality and yield of biodiesel. The properties of biodiesel met the quality standard of SNI 04-7182-2006. However, there was an increase in the yield of biodiesel from 42.73 into 49.34% and a decrease in the disposed methanol from 42.37 into 32.89%.

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