Biodiesel production with continuous processing and direct Ultrasonic Assisted

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Biodiesel Production with Continuous Processing and Direct Ultrasonic Assisted

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I. INTRODUCTION

The energy needs, especially the petroleum energy was increasing everyday.The world's crude oil total production amounted to 89.33 million barrels/day in third quarter of 2012 and the total consumption reached 90.08 million barrels/day[1]. This situation was affected nergy crisis. In Indonesia, the petroleum total consumption was amounted to 1.2 million barrels/day of crude oil during 2009 but the total supply just only 1 million barrels/day[2]. If energy comsumption was more than supply of energy, affected energy crisis in Indonesia.

This condition was wanted development of alternative fuel that low-costly, abundant raw materials, and easy to obtain from natural resources. Biodiesel is one of the appropriate alternative energy solutions. Biodiesel is a methyl ester that produced by esterification or tranesterification from vegetabe oil. The use and development of biodiesel was expanded in many countries during recent years[2][3][4].

Indonesia Country has a lot of natural resources that can be used as raw materials for biodiesel production. It was such as palm oil, soybean oil, olive oil, Nyamplung oil, rubber seed oil, etc. However, utilization of edible oil as a raw materials for biodiesel production was not recommended. It was affected in supply demand in food. So that it needed a source of vegetable oil that still have low using value, one of which is waste cooking oil [4][5].

The using of waste cooking oil as a raw material for biodiesel production can reduce the problem of waste in households, restaurants, and food processing industries. In Indonesia, the waste cooking oil generated by households, food processing industries and estaurants. Households have 305 thousand tons/year, the food processing industries have as much as 2 million tons/year and hotel and restaurant are 1.5 million tons/years. The total of waste cooking oil was available 3.8 million tons per year [6]. It was potential for utilization of waste cooking oil as raw material biodiesel production.

The recent technology that can be applied to produce biodiesel is the ultrasonic-assisted technology. Ultrasonic waves is the sound waves at 16 kHz - 10 MHz frequency. On the production of biodiesel, which is used is low frequency waves in range of 20-50 kHz [3][7]. Ultrasonic waves are contacted in the fluid will cause the microwaves (microbuble) or cavitation. After microbuble exploded, will formed hotspot that can increase the rate of transesterification for their hot spots that cause a rise in temperature [7][8][9]. The use of ultrasonic waves takes much faster than conventional technologies. Choedkiatsakul et.al., [7], research about production of biodiesel from palm oil. They was used ultrasound assisted reactor provided high methyl ester yield in only 25 minutes reaction time, whereas the mechanical stirred reactor required 60 minutes. Widayat et.al was produced biodiesel with ultrasonic assisted in 30 minutes and reached of biodiesel yield more 96% [2][3][4].

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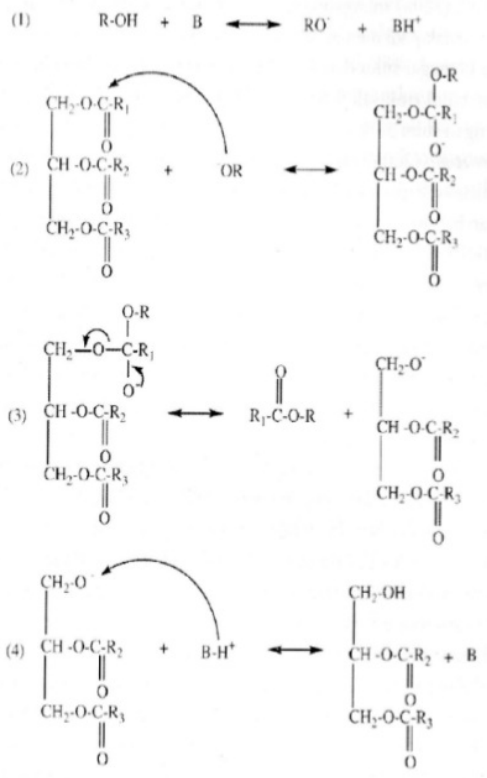


B : General catalyst
R_1,R_2,R_3 : Carbon chains of fatty acids
R : Alkyl alcohol

Fig. 1. Transesterification Reaction Mechanism with Base Homogeneous Catalysts

Waste cooking oil was conained free fatty acid (FFA) and triglyceride. FFA contents was very high and more 5%. In biodiesel production, waste cooking oil transesterified with methanol. The catalyst used in this research is homogeneous alkaline catalyst such as potassium hydroxide (KOH)[3][4]. Transesterification reaction mechanism with a homogeneous alkaline catalyst consists of four phases: (1) base catalyst form active species of RO-, (2) RO- nucleophilic attack the carbonyl group in triglyceride to form tetrahedral intermediate, (3) the intermediates are split to produce monoester, (4) Regeneration of active species RO-. These steps occur in R2 and R3 too [8].

Although base catalyst proved to be very efficient and popular in transesterification process but this catalyst does not show good results when the raw material has a moisture content and FFA contents ≥ 3%[4][9][10]. Therefore it takes a pretreatment to reduce the ammount of FFA in the waste cooking oil. This research was compared neutralization and esterification process for waste cooking oil pretreatment.

II. Methodology

Waste cooking oil was used as raw material that obtained from Banyumanik market, Semarang Central Java. Waste cooking oil was analyzed of free fatty acid. Potassium hydroxide (KOH) as a catalyst has analytical specification (Merck). Methanol has industrial specification. Biodiesel production carried out in reactor with capacity of 6000 ml. Ultrasonic wave was generated ultrasonic generator with a frequency of 30 kHz. This equipment is equipped with used frying oil tank and mixing tank. Mixing tank was used for methoxyde potasium preparation. The equipment's were presented in Fig. 2.

This research consists of two stages pretreatment and transesterification stage. Pretreatment was carried with neutralization and esterification reaction. Pretreatment of waste cooking oil for reduce the free fatty acids (FFA) content up to <3wt %[2][3]. Pretreatment was did in batch processing and ultrasonic assisted. The transesterification process was carried with a base catalyst continuously and ultrasonic assisted. Samples were taken every 10 minute and analyzed about viscosity and density. Products were separated by decantation and then washed to become a crude biodiesel. Biodiesel products were weighed to determine biodiesel yield. Crude biodiesel components are then analyzed using gas chromatography and analyzed its cetane number. Methods of this research is showed in Fig.. 3.

Transesterification was performed continuously using KOH catalysts and ultrasonic-assisted. Samples were taken every certain time and analyzed the density and viscosity, because of that response is one of the essential characteristics of biodiesel that can be observed its process dynamics. Then, the top layer of the product that is the crude biodiesel, were analyzed using gas chromatography mass spectrometry (GCMS) to know the content of its methyl esters.

III. Implementation and Result

A. *Pretreatment of Waste Cooking Oil*

Waste-cooking oil pretreatment by esterification reaction is using acid catalyst H_2SO_4 2 wt.% with ultrasound-assisted. Fig. 3 shown that all experiments are not met the value of < 3 wt.%. The lowest FFA value achieved at reaction time of 70 minutes that is 5 wt.%. There is no further action for this pretreatment because the condition aren't met yet.

Fig. 2. Biosonic reactor equipments.

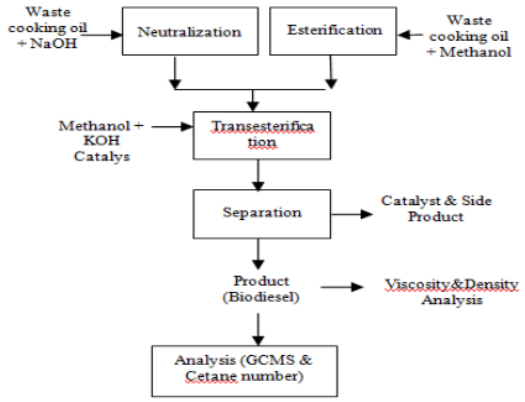


Fig. 3. Flow Chart of Research methods.

The other pretreatment is done by neutralization reaction method. Waste cooking oil is reacted with NaOH 18°C BE in various times. Ammount of NaOH that is used is limited according to the FFA contents that exist in the waste-cooking oil.

Fig. 4 shown that the lowest FFA content is achieved at 2.4 wt.% in 40 to 60 minutes. The FFA content in this method are met the requirements which is <3wt.% [10][11]. The optimum time for the neutralization reaction is 40 minutes for the FFA content met its requirements, while in the longer reaction time there was no significant change.

Neutralization reaction method was suitable for waste cooking pretreatment and effective to reduce FFA contents. This pretreatment need reaction time more less than esterification. Esterification is reversible reaction, so that if the condition of equilibrium has been reached then the reaction will be stable and may return to left side. To achieve 0.5% FFA content requires a longer time and stages of the process more than once.

Fig.. 6 is a corelation density versus time reaction in transesterification processing. It was shown that density of reaction product has decreased along with reaction time. It is

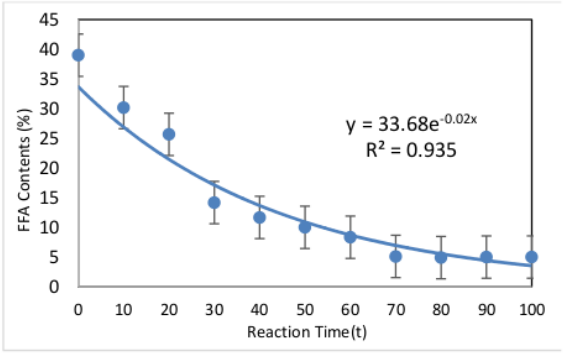


Fig. 4. FFA contents reduction on Waste-Cooking Oil Pretreatment with the esterification reaction.

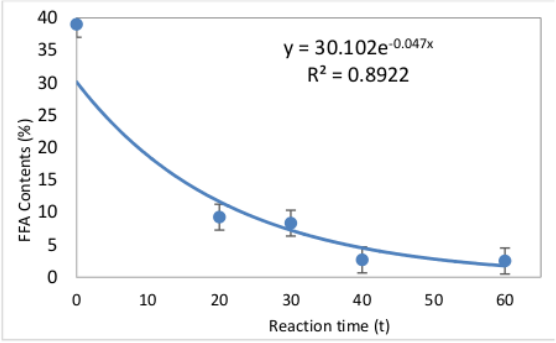


Fig. 5. FFA contents reduction on Waste-Cooking Oil Pretreatment with the neutralization reaction.

reaching a stable condition in 40-60 minutes. This indicates that triglyceride had begun converted into FAME / biodiesel. The density reduction phenomenon close to the density of biodiesel which is 0.850 to 0.890 g/ml. This value has similar with biodiesel standard in density parameter[2][3][10].

Transesterification is reversible reaction[2],[3]. In this research, methanol reactant was more excess than triglyceride reactant. This is affeceted in transesterification that will be close to irreversible reaction[15]. If the reactants have been no changes, then there was no reaction or stable condition in dynamic. This condition can be described with no decrease in the reactants or change parameters. It can also be observed from the phenomenon of density and viscosity changes. Density and viscosity of a mixture is a mixture of components in the mixture[3].

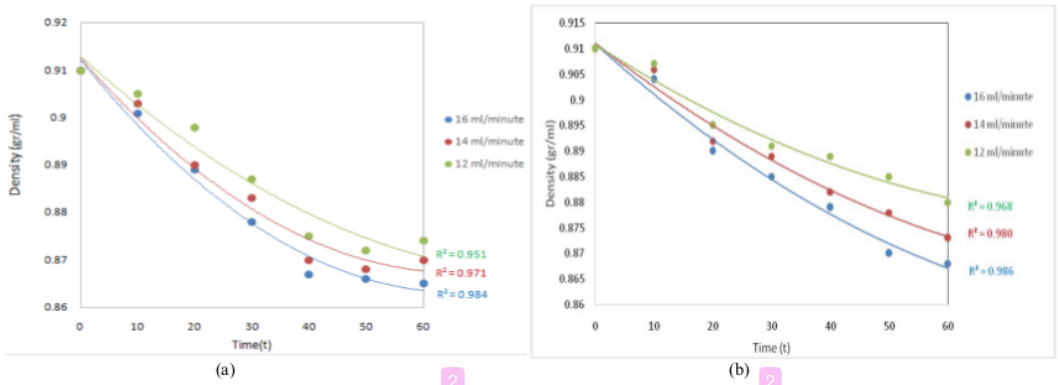
B. The Effect of Reactant Molar Ratio (Methanol : Triglycerides) to the Product's Density

Reactant molar ratio of 8:1 and 4:1 does not show a significant difference to density reduction that occurs, but the variable with a molar ratio of 8:1 at the time of ± 40 minutes, the product density has been in steady condition, while the molar ratio of 4:1 yet happens. This shows that the molar ratio of 8:1 has more quickly in steady state because it has excess methanol. This transesterification reaction is an equilibrium reaction wherein one reactant is made to be excess to drive the reaction towards the product.

So, it is known that the molar ratio of 8:1 is in steady state condition at the times of ± 40 minutes, and the molar ratio of 4:1 hasn't reached its steady state condition even it is at 60 minutes [4].

C. The Effect of Reactant Flow Rates to the Product's Density.

Fig. 5 also shown that the flow rate of 16 ml/minutes has the highest decrease of the density. This is because the greater flow rate, the more reactants going into the reactor too. This also cause the reaction is shifting toward to the product is much more, and the rate of reactant decomposition (dC_A/dt) is greater too. This is in accordance with the equation :



(a) (b)

Fig. 6. Density profile in continously transesterification reaction: (a) Molar ratio methanol to triglyceride 8:1, (b) Molar ratio methanol to triglyceride 4:1

$$\frac{dC_A}{dt} = \frac{F}{V}(C_{Ao} - C_A) - VC_A k_o e^{E_A/RT} \quad (1)$$

That the flow rate (F) is proportional to the value of the reactant decomposition (dC_A/dt)[12]. With a greater density reduction in the flow rate of 16 ml/minutes then the resulting product obtain the similarity with biodiesel's characteristics in a shorter time, than to other flow rate.

D. The Effect of Reactant Molar Ratio (Methanol: Triglycerides) to the product's Viscosity.

Fig. 7 is profile of fluid viscosity biodiesel product in transesterification reaction. The viscosity has decreased along with reaction time until was reaching a stable condition. Reactant molar ratio of 8:1 and 4:1 does not show a significant difference to viscosity reduction that occurs, but the variable with a molar ratio of 8:1 at the time of ± 40 minutes, the product viscosity has been in steady condition, while the molar ratio of 4:1 yet happens. This shows that the molar ratio of 8:1 has more quickly in steady state because it has excess methanol. This transesterification reaction is an equilibrium reaction wherein one reactant is made to be excess to drive the reaction towards the product [4].

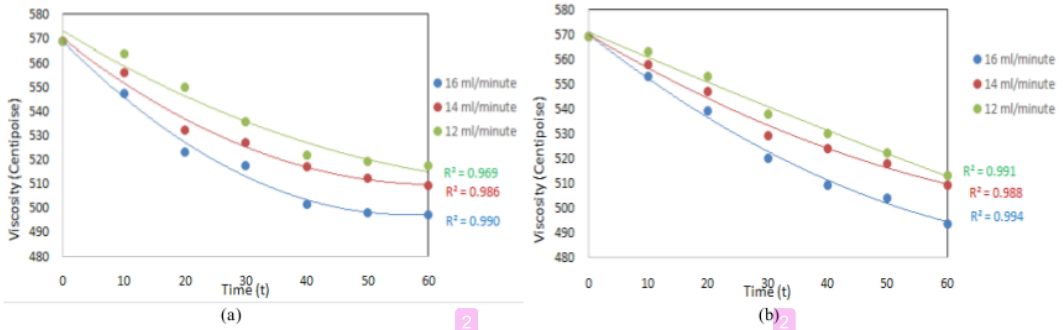
E. The Effect of Reactant Flow Rates to the product's Viscosity

Fig. 7 also known that the flow rate of 16 ml/minutes has the highest decrease of the viscosity. This is because the greater flow rate, the more reactants going into the reactor too. This also cause the reaction is shifting toward to the product is much more and forms methyl esther, which can be identified by a decreasing in viscosity.

This viscosity decreasing phenomenon occurs due to the density decreasing phenomenon too[13]. With a larger decrease in the flow rate of 16 ml/minutes then the resulting product obtain the similarity with biodiesel's characteristics in a shorter time, than to other flow rate.

F. Biodiesel's Yield

The effect of the molar ratio methanol to triglyceride to yield biodiesel is illustrated in Fig. 8. The highest yield for the molar ratio of reactants 8:1 and 4:1 are obtained at the same flow 16 ml/min with a value of 78% and 76,8%. The excess ammount of methanol in the transesterification reaction is essential to break the long-chain ester bonds into shorter-chain



(a) (b)

Fig. 7. Viscosity profile in continously transesterification reaction: (a) Molar ratio methanol to triglyceride 8:1, (b) Molar ratio methanol to triglyceride 4:1

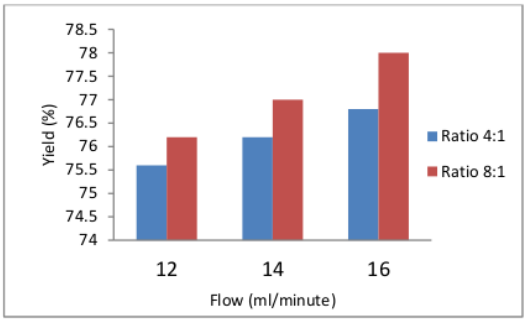


Fig. 8. Biodiesel yield of continous processing.

TABLE I. BIODIESEL CHARACTERISTICS

No	Parameter	Units	Results	SNI 04-7182-2006
1	Cetane Number	-	51,2 – 57,3	Min. 51
2	Density	Kg/m^3	865 – 880	850 – 890
3	Acid Value	Mg-KOH/g	0,58 – 0,72	Maks. 0,8
4	Viscosity	cSt	5,7 – 5,9	2,3 – 6,0

ester which is methyl esther [14]. In this research, the highest yield of methyl ester is obtain at reactant molar ratio 8:1 due to the amount of methanol required in the transesterification reaction is sufficient to break long-chain ester bonds into shorter chain esters.

IV. CONCLUSION

Biodiesel was produced from waste cooking oil with continous processing and ultrasonic assisted. Waste cooking oil has FFA contents more than 0.5%, so must pretreatment for reduction this. The best method of pretreatment waste cooking oil is neutralization with sodium hydroxide that need reaction time about 40 minute. Biodiesel production that carried on molar ratio methanol to oil reactant 8:1 and flow rate 16 ml/minutes, has better than molar ratio methanol to oil 4:1. Biodiesel products had characteristic with Indonesia biodiesel standard. Yield of biodiesel maximum obtained about 78%.

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