Pertanika J. Sci. & Technol. 21 (2): 567 - 580 (2013)

PERTANIKA JOURNALS

SCIENCE & TECHNOLOGY

Journal homepage: http://www.pertanika.upm.edu.my/

Palm Oil Transesterification Processing to Biodiesel Using a Combine of Ultrasonic and Chemical Catalyst

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ABSTRACT

A combination of ultrasonic and chemical catalyst affects to the transesterification reaction rate of palm oil conversion to biodiesel is investigated in a 5 Liter capacity reactor equipped with the heater and temperature controller and the 42 Hz ultrasonic generator of 35 watt with process parameters of (a) reaction temperature, (b) methanol to palm oil ratio, (c) and the amount of chemical catalyst and (d) duration of the ultrasonic activation. The result shows that by combining the ultrasonic and chemical catalyst in the palm oil transesterification processing increases the chemical reaction rate, the higher the frequency of the ultrasonic activation used, the higher the biodiesel conversion.The transesterification operating process condition of 60 to 70 ^{o}C, Methanol to palm oil ratio of 5, reaction duration time of 60 minute, and catalyst activation of 30 minute, using a 42 KHz , 35 watt ultrasonic, may be applied to produce biodiesel with a conversion as high as 94% for palm oil feed stock and 91% for coconut oil feedstock

Keywords: Biodiesel, palm oil, transesterification, ultrasonic, chemical catalyst

INTRODUCTION

The surging price of cure oil, together with environmental issue has been encouraging many countries to seek alternative fuels. Since 2004, Indonesia becomes a net importer of crude oil. Some alternatives energy sources have been developed, such as Gas and coal, Hydropower, Geothermal and Biofuel. Two well-known biofuels commercially produced are biodiesel and bioethanol. As a large agricultural country, Indonesia has very big potential to produce biofuel. Currently, most biodiesel is commercially produced from vegetable oils by transesterification / esterification reaction. This is the most common process technology of biodiesel that proven in the world.The detailed process of biodiesel production varies between one another depending on licensor. However, the

Article history:
Received: 26 December 2011
Accepted: 15 March 2012

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ISSN: 0128-7680

principle of process is similar which consisting of esterification transesterification reaction followed by glycerol separation and then ester (biodiesel) purification.

The Indonesia domestic consumption of fuel oil has been increasing steadily, while the production of fuel oil and its availability tends to decrease. The continued reliance on fossil fuel for fulfilling national energy demand has to be gradually decreased by utilizing other energy sources including agricultural products. Developing biofuel is a must for Indonesia due to several reasons including the abundance availability of feed stocks, its potential to improve air quality in big cities and its potential to reduce the poverty in rural areas. Two well-known biofuels commercially produced are biodiesel and bioethanol. In the year 2010, petroleum solar oil consumption in Indonesia is about 28 million kilo Liters and petroleum diesel oil is more than 300 kilo Liters. This means that for 10% of biodiesel mixture with 90% petroleum solar or diesel oil (B10), there will be a demand of more than 2.8 million tons of biodiesel per year in 2010. This is huge demand for biodiesel in Indonesia (Biodiesel Research Group, 2007). The technology for producing biodiesel from vegetable oil need to be developed and improved. The use of combining of the ultrasonic and chemical catalyst is introduced in this paper.

SELECTION OF BIODIESEL FEEDSTOCK

The advantage of biodiesel over fossil fuel is its sulfur free, so that its flue gas can be easily controlled and environmentally friendly. The Kyoto Protocol signed in 1997 requires, from the European Union (EU), a commitment to reduce, between 2008 and 2012, greenhouse gas emissions by 8% from their 1990 level. One of the instruments chosen by the EU Commission to achieve these results is reported in the Directive 2003/30/EU, which states that, starting from 2010, at least 5.75% of the fuel used for driving engines in a number of EU states should come from renewable sources. However, it is well-known that biodiesel must be mixed with diesel or solar oil if it is to be used for combustion engine, with the usage range from 5%(B5) to 40% (B40) of biodiesel. Although 100% of biodiesel can also be used directly to the engine but this is not common practice. Problems associated with the use of vegetable oils as fuel were oil deterioration and incomplete combustion. Polyunsaturated fatty acids were very susceptible to polymerization and gum formation caused by oxidation during storage or by complex oxidative and thermal polymerization at the higher temperature and pressure of combustion. The gum did not combust completely, resulting in carbon deposits and lubricating oil thickening. To overcome these problems the oil requires slight chemical modification mainly transesterification, pyrolysis and emulsification. Among these, the transesterification is the key and foremost important step to produce the cleaner and environmentally safe fuel from vegetable oils (Biodiesel Research Group, 2007).

Currently, most biodiesel is commercially produced from vegetable oils by transesterification / esterification. This is the most common process technology of biodiesel that proven in the world. In the reaction, the oil and alcohol are reacted to produce alkyl esters (biodiesel). Methanol is used as the alcohol for commercially producing biodiesel because it is the least expensive alcohol. Hence, methanol is important raw material for biodiesel, with its demand in Indonesia being around 280,000 ton per year.

Process technology of a biodiesel plant depends upon the quality of the feedstock, the plant capacity and the quality requirements for finished biodiesel. It is well known, transesterification or esterification is an equilibrium reaction. To ensure the increase of triglyceride conversion, an excess of alcohol is used. The minimum equivalent ratio of alcohol to vegetable oil is three.

The biodiesel research conducted in Gadjah Mada University Yogyakarta Indonesia was started in 1980's. The vegetable oil processed were rubber seed oil, peanut oil, castor oil, nyamplung seed oil, jatropha curcas oil, palm oil, kapuk seed oil, and candlenut oil etc. The equivalent ratio of alcohol (ethanol or methanol) to vegetable oil is in the range of 3 to 14. The higher the equivalent ratio of alcohol to vegetable oil, the higher the conversion is, but the cost of alcohol recovery is also higher. An excessive amount of alcohol also makes the recovery of glycerol difficult, so the optimum ratio of alcohol to oil has to be established, considering each individual process (Biodiesel Research Group, 2007; Supranto, 2003, 2004)). Some researchers (Supranto, 2004; Freedman *et al*., 1986; Encinar *et al*., 2005) found the molar ration of 6 : 1 of methanol to oil gave the best conversion. Moreover Freedman *et al*. (1984) studies the effect of molar ratio on ester conversion with vegetable oils such as soybean, sunflower, peanut and cotton seed oils behaved similarly and achieved highest conversions (93 – 98%) at 6 : 1 molar ratio. The similar results have been found for transesterification of rubber seed oil with methanol (Mustanginah, 2006).

CPO is recently verry attractive to be converted to biodiesel, although CPO based biodiesel is a bit unsuitable in European countries. Other potential raw materials for biodiesel are abundant in Indonesia, including coconut oil and Jathropa Curcas. Until 2005, increasing planting area was observed not only in Jatropha but also Oil Palm and Peanut. Increasing oil palm planting area is due to the world demand for oil palm edible oil that increases every year and there is enough suitable land across the country (Supranto, 2010).

A. Palm Oil

Crude palm oil can be processed to produce many product derivatives which are very important in supporting different types of industry. Indonesian government has targeted to enlarge oil palm area till 18 millions tons in 2010. The oil palm planting area since 2000 has been spread out in Sumatra, Kalimantan, Sulawesi and Papua. Until 2005, North Sumatra and Riau dominate the area in the country in almost 1 million ha each. Riau has the largest oil palm area in the country, though the local production in North Sumatra is higher. The productivity of oil palm in North Sumatra is the best across the country. The production can reach 3785 ton/ha/year of bunch (Biodiesel Research Group, 2007).

B. Coconut

The use of oil palm to produce edible oil has affected the development of Coconut planting area. Though the total number of Coconut planting area increased in 2001, the planting area then has continually decreased till 2005. The reduction of Coconut planting area might be due to its cultivation being not economically visible. Many estates have changed their Coconut to other profitable estate crop such as oil palm. Riau, Central and East Java, North Sulawesi are the area of coconut. Coconut can be found in the back yard and dry land field especially in the

coastal side. Now days, coconut was cultivated extensively across the country, therefore the productivity seems very low. In the centre production area, the productivity can reach only 1 ton/ha. The best productivity was reported in Central Sulawesi, the highest local production was observed in Riau. The potential production of coconut is estimated to be stagnant until 2010 (Biodiesel Research Group, 2007).

C. Jathropa

Jatropha was uncultivated crop which is easily founded in the fence of field or backyard. Jatropha can be grown in different types of land. In the marginal land such as unfertile dry land area, it can produce enough seed. In the national level, the planting area of jatropha in 2000 was only 12807 ha. Lampung, Java, Nusa Tenggara and Central Kalimantan were the place where jatropha was easily seen. East Java and Nusa Tenggara which were believed as dry area have the largest planting area. Since 2000, the planting area in Lampung, Banten, Central Java, East Java and West Nusa Tenggara has no significant increased. Different situation could be seen in West Java and East Nusa Tenggara. The planting area has significantly inclined. In 2005 the planting area has 10 times multiplied. The trend of increasing planting area in West Java and East Nusa Tenggara may be due to the concern of the government in developing energy source diversification. In West Java, several research institutions have proposed jatropha as energy source alternatives, therefore many field researches may be conducted in the area. Field action research may also been executed in East Nusa Tenggara due to the dry climate in the area (Biodiesel Research Group, 2007).

Increasing planting area has not been followed by the increase of seed production. Different effect can be observed between West Java and East Nusa Tenggara. Though the productivity in these two areas right now is considered very low, West Java can maintain its productivity. Different result has been founded in East Nusa Tenggara; increasing planting area may even reduce the productivity. This phenomenon might be caused by the condition that the cultivation technology was still missing. The potency to increase national seed production is still possible because there is a very large suitable area and government support policy.

Indonesia has very big potential to produce biodiesel doe to the fact that Indonesia is the second largest Crude Palm Oil (CPO) producer in the world and also has a very large area of Coconut plantation. About 75% of the product CPO is exported as crude material. CPO is recently the most attractive followed by the Coconut oil to be converted to biodiesel, although CPO based biodiesel is a bit unsuitable in European countries. The development of technology for producing biodiesel from Palm and Coconut Oil are need to done such as by combining the use of ultrasonic and chemical catalyst to increase its rate processes.

EXPERIMENTAL RESULT AND DISCUSSION

A. Experimental Work

The objective of experimental work of converting palm and coconut oil to biodiesel using a combine ultrasonic and chemical catalyst was to identify the suitable process condition,

including temperature, ratio of the methanol (MET) onto the Tri Gliceride (TGE) in the Palm Oil (PO) and Coconut Oil (CO) through a transesterification process. The product of the complete transesterification are Fatty Acid Methyl Ester (FAME) as the main product and Glycerol (GLY) as the bye-product. The chemical process was carried out in a batch reactor system equipped with the heater and temperature controller and the 42 Hz ultrasonic generator of 35 watt to activate the mixture of methanol and NaOH catalyst. The process parameter inverstigated are (a) reaction temperature , (b) methanol to palm oil ratio, (c) amount of chemical catalyst and (d) duration of the ultrasonic activation.

The experimental apparatus consist of a 5 Liter capacity batch reactor, equipped with heater, sampling system and the recirculating pump as the mixing part. The ultrasonic compartment is set outside the reactor, in the stream line of the methanol-catalyst mixture to reactor inlet..

The experiment was carried out with variables of process condition including: temperature from 30 to 70 ^{o}C; ratio of MET/TGE on Palm and Coconut Oil of 3 to 6; NaOH Catalyst of 0.25 to 1 % and the duration of catalyst activation by the ultrasonic part of 5 to 30 minutes. In all of the experimental works, the ultrasonic generator used was having properties of 42 KHz frequency with 35 watt power supply.

B. Experimental Result

The result of the experiment are presented in Fig.1 to Fig.4, showing the effect of the variation of temperature, variation of the ratio of MET/TGE, variation of the amont of NaOH catalyst, and variation of the duration of catalyst activation, on the convertion of TGE in Palm and Coconut Oil respectively.

Fig.1 shows the effect of temperature and time duration on the Palm and Coconut TGE conversion .As shown in Fig.1, the TGE conversion is increasing when longer duration time is used, however the increased of TGE conversion will no longer significant when the duration reaction time reachs 60 minutes. In general it can be seen in Fig.1, that the transesterification of TGE Palm Oil is faster than that of Coconut Oil. This may be chemical component in the TGE Palm Oil has higher activity than that in the TGE Coconut Oil.

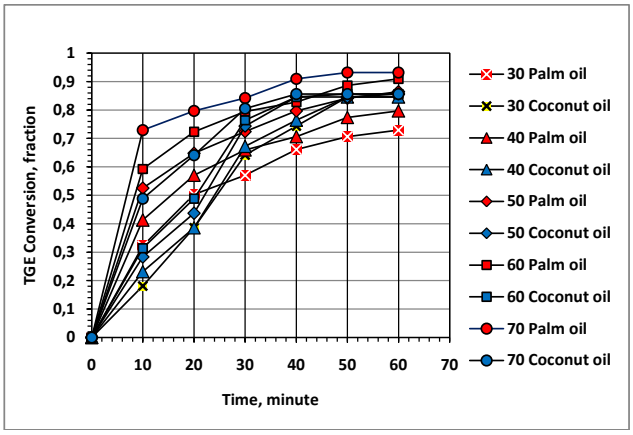

Fig.1: The effect of temperature and time duration on the Palm and Coconut TGE conversion with the MET/TGE ratio of 4, NaOH catalyst of 1% and ultrasonic activation of 30 minutes

The higher the reaction temperature is used, the faster the reaction rate, however the reaction temperature is not able to be increased higher than 70 °C doe to the normal boiling point (65°C) property of pure methanol reactant used.

Fig.2 shows the effect of varying mole ratio MET/TGE on TGE conversion. It is shown in Fig.2, that in general, the transesterification of TGE Palm Oil is faster than that of Coconut Oil. Fig. 2 also shows the TGE conversion is affected by the MET/TGE ratio. Increasing the MET/TGE ratio from 3 to 6 will increased the TGE conversion of about 25% for Palm Oil, and about 60% for Coconut Oil. The increased of MET/TGE used will reduce the viscosity of mixture, which may cause better contact for the reactants and simultaneously increase the reaction rate.

Fig.3 shows the effect of catalyst and time duration on the Palm and Coconut TGE conversion. The combine of ultrasonic generation of 42 KHz frequency with 35 watt power supply and chemical of sodium hydroxide (NaOH) was used as catalyst in the TGE transesterification. The ultrasonic was used to activated the mixture of methanol and NaOH

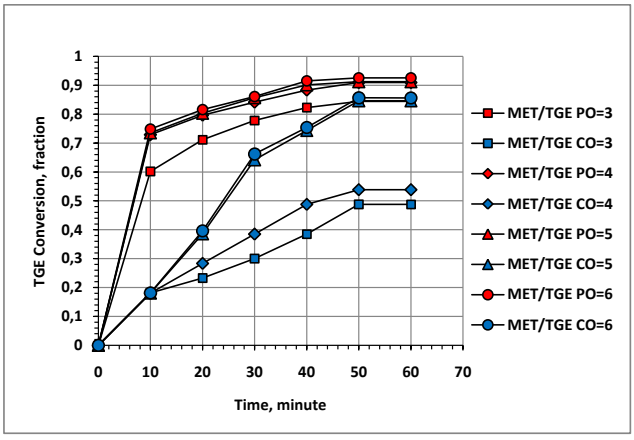

Fig.2: The effect of the MET/TGE ratio and time duration on the Palm and Coconut TGE conversion with the reation temperature of 70 °C, NaOH catalyst of 1% and ultrasonic activation of 30 minutes.

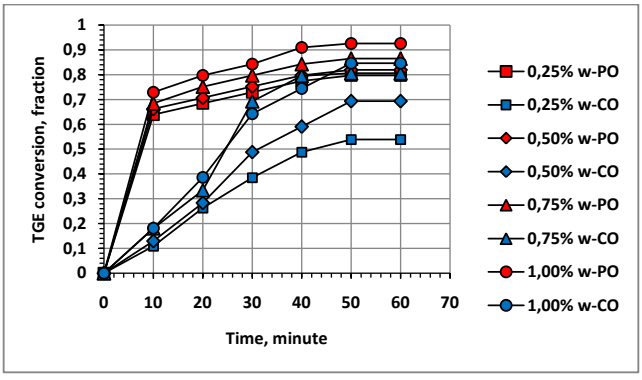

Fig.3: The effect of catalyst and time duration on the Palm and Coconut TGE conversion with the reation temperature of 70 °C , the MET/TGE ratio of 5, NaOH catalyst of 1% and ultrasonic activation of 30 minutes.

catalyst. It is shown in Fig.3 that by increasing the amount of NaOH catalyst, the reaction rate of transesterification is also increasing. By using the low amount of NaOH catalyst i.e. 0.25%, the difference of TGE Palm Oil and Coconut Oil Conversion is higher than that by high amount catalyst, i.e. 1% . It seems that a suficient amount of chemical catalyst is needed in this transesterification process.

The effect of ultrasonic activation on the methanol-catalyst mixture is shown in Fig.4. The result of the experiment shows that by combining the ultrasonic and chemical catalyst in the palm and coconut oil transesterification processing increases the chemical reaction rate. The optimal conversion of the the palm oil transesterification can be achieved at 60 minute transesterification process using the process condition of the temperature of 70°C, the methanol to palm oil ratio of 4, the catalyst amount to palm oil of 1% (w/w), and the 42 KHz ultrasonic activation periode of 30 minutes.

C. Transesterification Kinetic Evaluation

The chemical reaction of transesterification is simplified as shown in Equation [1], with MET is methanol, TGE is triglyceride in Oil, FAME as the main product (biodiesel) and GLY is glycerol as the bye-product. The chemical reaction kinetic is approach as a second order of pseudo homogeen reaction, with the reaction rate formula is shown in Equation [2] and simplified to Equation [3] with x representing the TGE conversion, k is the coefficient of the reaction rate, C_{TGE0} is the initial TGE concentration and t representing the duration time of the reaction.

$$\underset{3\ \text{mole}}{\text{MET}} + \underset{1\ \text{mole}}{\text{TGE}} \xrightarrow{k} \underset{3\ \text{moles}}{\text{FAME}} + \underset{1\ \text{mole}}{\text{GLY}} \qquad [1]$$

MET: Methanol, TGE: Three Glyceride, FAME: Fatty Acid Methyl Ester (biodisel), GLY: Glycerol

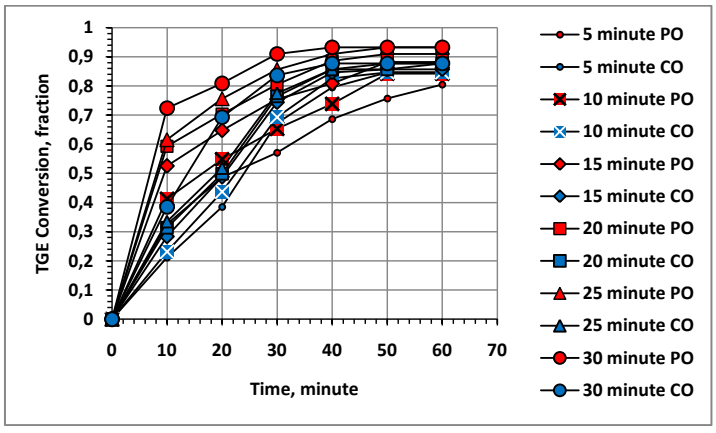

Fig.4: The effect of ultrasonic activation time duration on the Palm and Coconut TGE conversion with the reation temperature of 70 °C, the MET/TGE ratio of 5 and NaOH catalyst of 1%.

$$-r_{TGE} = k\ C_{TGE}^2 \quad [2]$$

$$dx/dt = k\ C_{TGE0}\ (1-x)^2 \quad [3]$$

The value of k for each set data of the experimental result can be determined which gives the smallest sum square error. The value of k determined from experimental result and Equation [3] can be used to calculate or predict the value of TGE conversion based on a second order of pseudo homogeen reaction approach fitted to the experimental data, as described by Supranto (2010). The result of the kinetic evaluation are presented in Fig.5 for the variable of reaction temperature, Fig.7 for the variable of NaOH catalyst amount, and Fig.9 for the Variable of ultrasonic activation duration time. The coefficient of reaction rates determined from the kinetic evaluation and experimental result then plott in Fig.6 for the temperature variable, Fig.8 for the amount catalyst variable and Fig.10 for the variable of ultrasonic activation time.

Fig.5 is fit to represent the experimental data as shown in Fig.1. The coefficient of transesterification reaction rates(k) determined from the kinetic evaluation and experimental result in Fig.1 are presented in Table 1 and Fig.6.

As shown in Table 1 and Fig.6, the Coefficient of reaction rates of Palm Oil TGE transesterification is higher than these of Coconut Oil TGE transesterification. The length of carbon chain and type of chemical, having double bound or none in the raw material Palm Oil and Coconut Oil may affect in transesterification of Palm Oil and Coconut Oil.

TABLE 1
Coefficient reaction rate of TGE transesterification

Temperature, °C	.k, g/mgrek.min	
	Palm Oil	Coconut Oil
30	0.0157	0.0118
40	0.0211	0.0127
50	0.0325	0.0154
60	0.0422	0.0169
70	0.0771	0.0245

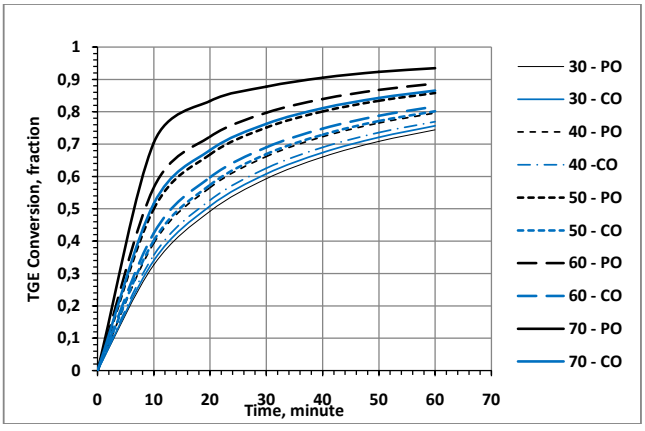

Fig.5: The Calculated Palm and Coconut Oil TGE conversion base on a second order of pseudo homogeen reaction with parameters of the reaction temperature and the reaction duration time.

Fig.7 is fit to represent the experimental data as shown in Fig. 3. The coefficient of transesterification reaction rates(k) determined from the kinetic evaluation and experimental result in Fig.3 are presented in Table 2 and Fig.8.

TABLE 2
Coefficient reaction rate of TGE transesterification

NaOH catalyst amount	% of NaOH catalyst	
	Palm Oil	Coconut Oil
0.250	0.0357	0.0035
0.500	0.0408	0.0048
0.750	0.0523	0.0070
1.000	0.0746	0.0071

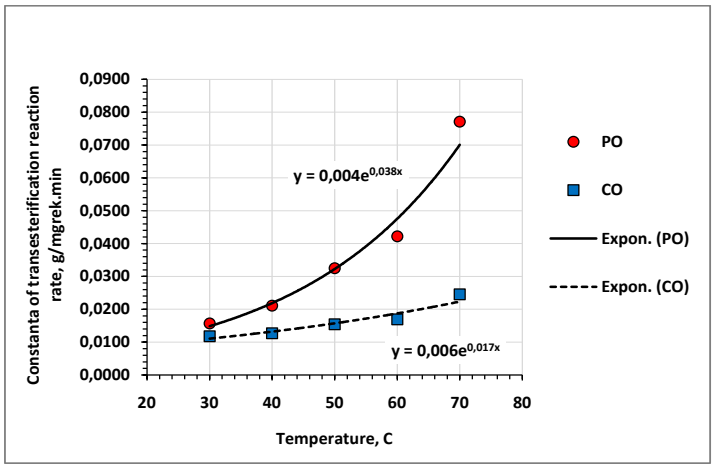

Fig.6: The effect of reaction temperature on the coefficient of reaction rates of TGE transesterification.

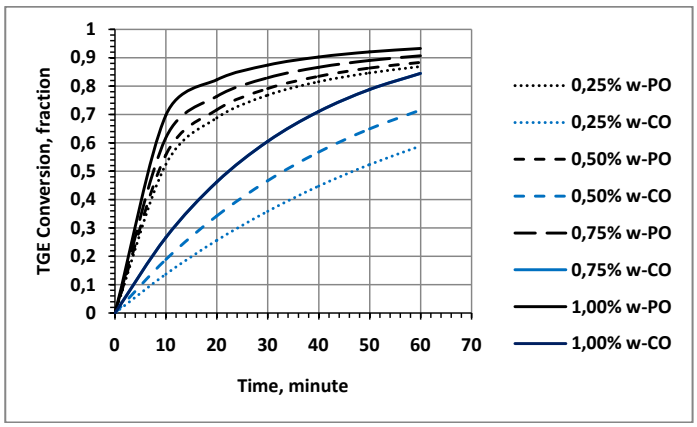

Fig.7: The Calculated Palm and Coconut Oil TGE conversion base on a second order of pseudo homogeen reaction with parameters of the NaOH catalyst amount and the reaction duration time.

The coefficient of transesterification reaction rates determined from the kinetic evaluation and experimental result on ultrasonic activation time are plotted in Fig.9. It is shown in Fig.9 that the TGE conversion increasing when ultrasonic activation time increasing. The transesterification reaction coefficient affected by the ultrasonic activation is shown in Fig.10.

The corelation of the ultrasonic activation duration time and the transesterification reaction rate coefficient is shown in Fig.10. The corelation is a logaritmic type.

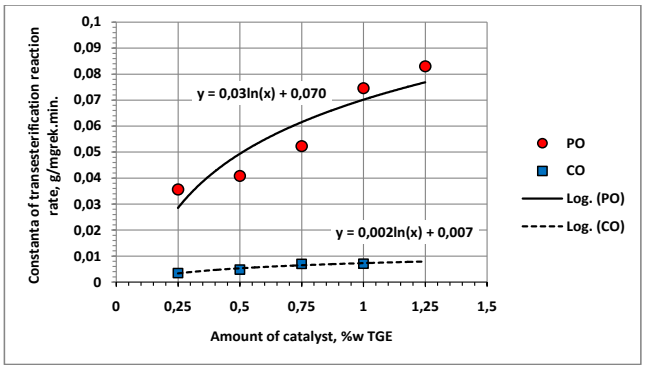

Fig.8: The amount catalyst effect on the coefficient of reaction rates of TGE transesterification.

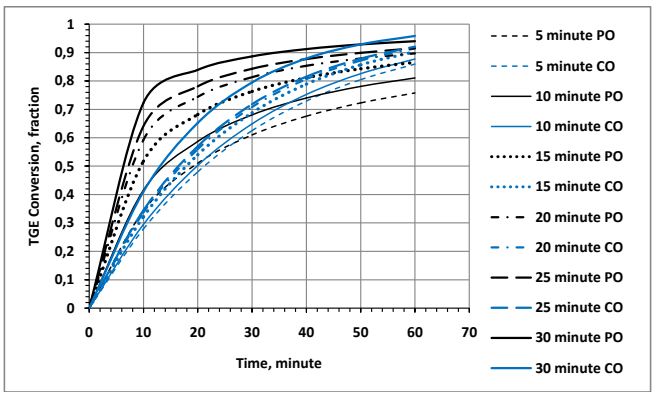

Fig.9: The Calculated Palm and Coconut Oil TGE conversion base on a second order of pseudo homogeen reaction with parameters of the ultrasonic catalyst activation and the reaction duration time.

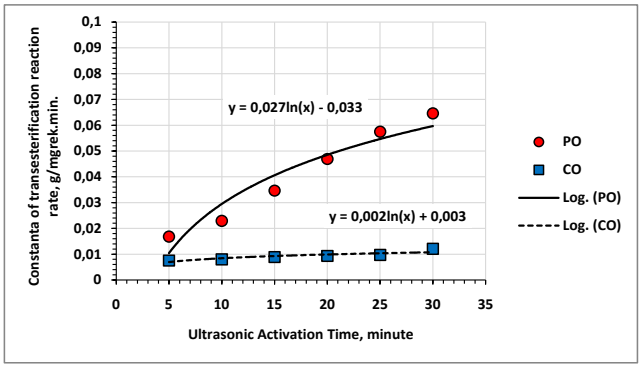

Fig.10: The effect of ultrasonic activation duration time on the coefficient of transesterification reaction rates.

D. Ultrasonic Frequency

Some experimental result of transesterification using ultrasonic activation of 40 KHz to 600 KHz have been found in publish literature, as shown in Table 3. Jose *et al.* (2005) using Soybean Oil (SO) as feedstock, with ultrasonic of 80 to 100 KHz, power of 120 watt, in a batch type reactor have reached TGE conversion of 97.5%. Vishwanath *et al*. (2008) using Palm Fatty Acid Distillate (PFAD) as feedstock, with ultrasonic of 50 KHz, power of 120 watt, in a batch type reactor have reached TGE conversion of 94%, while Mahamuni *et al.* (2009) have reached a TGE conversion of 98.5 when using Soybean Oil as feedstock, with ultrasonic of 323 to 611 KHz,and power of 323 watt.

Without taking the differences in feedstocks, catalysts, reactant ratios, temperatures, reactor dimensions and other operating conditions into consideration, the effect of ultrasonic frequency on the TGE conversion may be figurized as shown in Fig.11. It is shown in Tabel 3 and Fig.11 that transesterification process assisted by an ultrasonic with frequency of higher than 100 kHz promises a TGE conversion of higher than 97.5%, which is a very high conversion of chemical process.

TABEL 3
Transesterification using ultrasonic freq. 40 to 300KHz

Researcher	Jose *et al.* (2005)	Vishwanath *et al.* (2008)	Mahamuni *et al.* (2009)	Author	Author
Feedstock	SO	PFAD	SO	PO	CO
Catalyst	NaOH	NaOH	KOH	KOH	NaOH
Catalyst %	1	1	0,50	1	1
MET/TGE	6	5	6	5	5
Temperature, C	25 – 60	30– 50	25– 50	30 – 70	30 – 70
Ultrasonic., KHz	80-100	50	323-611	42	42
Ultrasonic, watt	120	120	323	35	35
Reaction Order	2	-	2	2	2
Time, minute	60	60	60	60	60
Process type	Batch	Batch	Batch	Batch	Batch
Conversion, %	97.5	94	98.5	93	91

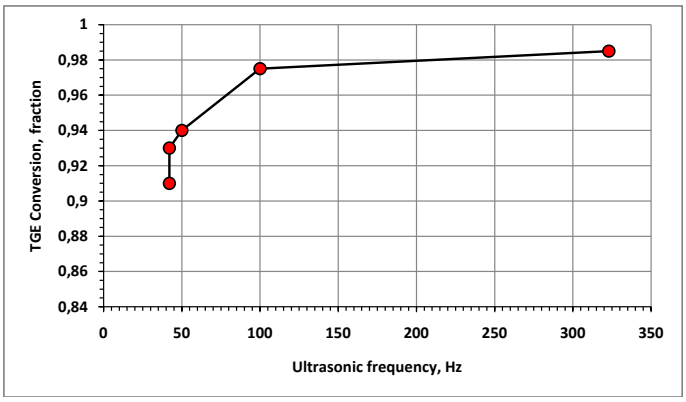

Fig.11: The effect of ultrasonic frequency on TGE conversion

CONCLUSION

From the experimental result and the literature study some conclusion may be sumerized as follow:

1. The transesterification process of Palm and Coconut Oil using methanol with an ultrasonic and chemical catalyst of NaOH follows a reaction kinetic of order 2. The reaction rate coefficient of palm oil transesterification is almost 10 time higher than that of coconut oil.
2. The corelation of the temperature and the transesterification reaction coefficient is an exponential type, while the ultrasonic activation and catalyst related to the transesterification reaction coefficient is of logaritmic corelation type.
3. The transesterification operating process condition of 60 to 70 ^{o}C, MET/TGE ratio of 5, reaction duration time of 60 minute, and catalyst activation of 30 minute, using a 42 KHz, 35 watt ultrasonic, may be applied to produce biodiesel with conversion as high as 94% for palm oil feed stock and 91% for coconut oil feedstock.
4. In general the higher the frequency of the ultrasonic activation, the higher the conversion of the TGE to bodiesel. Hovewer the use of ultrasonic frequency higher than 400 KHz is not recomended doe to a very high of electric energy consumption.

ACKNOWLEDGEMENTS

Author would like to thank to the Indonesia government c.q. the Ministry of research and technology, for the financial support of this research through an Insentif Research Program 2010 "*Pengembangan propelan berbasis material nabati*".

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