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Biodiesel Production from Palm and Waste Cooking Oils Catalyzed by Silica-Titania Catalyst with Time Variation

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Abstract:- The optimation of reaction time on biodiesel production from palm oil and waste cooking oil catalyzed by silica-titania has been investigated. The reaction time varied in the range of 2-6 hs. The optimum condition has been obtained by flow rate, density, and acid number analysis of biodiesel product. The results show that biodiesel product from palm oil has optimum reaction time of 4-5 h with percentage of conversion of 33.33%. The same optimum reaction time is also shown for biodiesel product from waste cooking oil, however, the percentage of conversion is higher, i.e. 57.14%. The higher reaction time (6h) yields lower percentage of conversion due to back reaction of hydrolysis of fatty acid ester.

***Keywords*:-** *Biodiesel; FAME; FFA; Percentage of Conversion; Silica-Titania Catalyst.*

I. INTRODUCTION

Biodiesel is one of alternative energy resources obtained from transesterification reaction between vegetable oil or lard fat and short chain alcohol using a given catalyst [1]. The vegetable oil sources commonly used in biodiesel production are corn oil, palm oil, soybean oil, and sunflower oil [2–5]. The superiority of biodiesel energy is that it is renewable, biodegradable, non toxic, sulfur free, releasing lower CO2 than that of petroleum oil, and friendly environmental [6,7]. Therefore, research on biodiesel product is continually developed with respect to oil resources, catalyst, and parameter being used.

The reaction time is one of the factors affected biodiesel production. Biodiesel from the same source of vegetable oil using different catalyst shows different reaction time. On the other hand, using the same catalyst shows different percentage of conversion if the reaction time is not the same. For example, biodiesel production from palm oil using egg skin catalyst requires reaction time of 2h to yield 94.1% biodiesel. The biodiesel production from soybean oil requires reaction time of 3h to yield 95% biodiesel using the same catalyst [8].

Silica-titania is a potential heterogeneous catalyst, which is used in biodiesel production through transesterification reaction. The application of silica-titania catalyst in biodiesel production has only a few reported. Among several variables in silica-titania catalyst used in biodiesel production only substitution of sulfate group has been reported [9]. In this research group, several investigations on the effect of calcinations temperature on the synthesis of silica-titania with solid-state method with respect to titanium tetrahedral fraction in SiO_2-TiO_2 catalyst, effect of mol ratio of SiO_2 : TiO_2 on titanium tetrahedral fraction in SiO_2 : TiO_2 catalyst in biodiesel production, effect of catalyst mass of SiO_2-TiO_2 in biodiesel production from palm oil and waste cooking oil using silica-titania catalyst have already reported [10–13]. However, the investigation on the effect of optimum reaction time in biodiesel production from palm oil and waste cooking oil in relation to physical properties and percentage of conversion has not yet been reported [11–13]. Therefore, this paper will report the variation of reaction time in biodiesel production from palm oil and waste cooking oil applying silica-titania catalyst.

II. MATERIALS AND METHOD

A. Materials

The synthesis of silica-titania catalyst requires commercial SiO_2 and TiO_2, and toluene (Merck). The materials for biodiesel production and acid number assay are palm oil (bimoli), waste cooking oil, methanol (Merck), n-hexane, KOH, ethanol, phenolphthalein indicator and aquadest [12].

The equipment for synthesis include glassware, hot plate, oven, furnace, balance, thermometer stirrer, centrifuge, ultrasonic, and rotary evaporator. The instruments applied for characterization of physical properties include picnometer, and FTIR.

B. Procedure

The silica-titania catalyst used was based on the optimum condition obtained from previous studies [11,12]. That catalyst was synthesized by mixing of 0.5 mmol TiO_2 and 1.00 mmol SiO_2 solids in 10 mL of toluene and continued by drying at room temperature to evaporate toluene as well as calcined at temperature of 450^{o}C for 8h [11–14].

The procedure of biodiesel production was carried out by following the procedure in the previous works [11,12]. Biodiesel was produced by tranesterification reaction between palm (bimoli) and waste cooking oils with methanol using silica-titania catalyst. The oil source and methanol were reacted at a ratio of 6 : 1 in a three bottle-neck Erlenmeyer (250 mL) with a reflux system provided

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by a thermometer (360°C) at 65°C for a variation of reaction time, i.e. 2, 3, 4, 5, and 6h. The reaction was conducted using the catalyst 7% by weight of oil source [13]. Afterwards, the catalyst was separated from the reaction product by centrifuge and the followed by removal of the excess methanol using rotary evaporator [13,14]. The product was analysis by several properties such as density, viscosity and acid number as well as characterized by FTIR (Fourier Transformation Infrared) at wave number of 600 – 4000 cm^{-1}.

Sample	Label	Sample	Label
Palm Oil	PO	Waste Cooking Oil	WCO
2 hours	BPO2	2 hours	BWC2
3 hours	BPO3	3 hours	BWC3
4 hours	BPO4	4 hours	BWC4
5 hours	BPO5	5 hours	BWC5
6 hours	BPO6	6 hours	BWC6

Table 1:- Table of Sample Label

III. RESULTS AND DISCUSSION

A. FTIR Spectra of Oil Source and Biodiesel Product

Fig. 1 show the FTIR spectra of biodiesel product with the highest percentage of conversion and FTIR spectra of oil sources (palm oil and waste cooking oil).

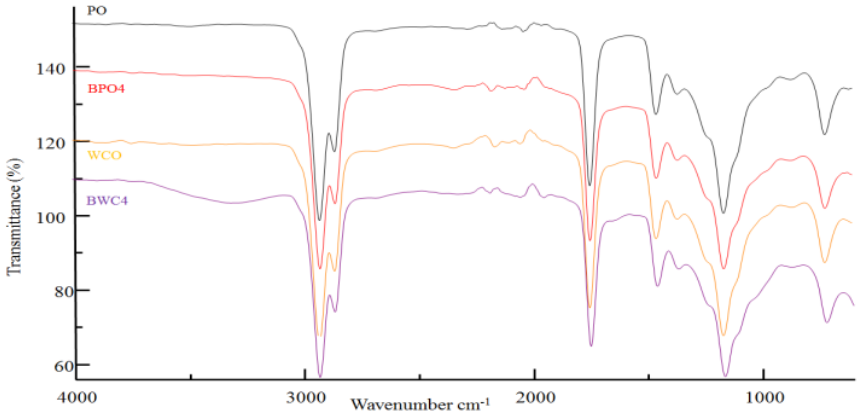

Fig 1:- FTIR spectra of oil source and biodiesel product.

On the basis of Fig. 1 the FTIR spectra of oil sources are generally similar with that of biodiesel products. However, the absorption band at wave number 1300 – 1000 cm^{-1} show differences in intensity. The absorption band in this range is due to vibration frequency of C-O-H and C-O-C from fatty acid ester or biodiesel. The shift of absorption band of C-O-C ester from oil sources to form a broader band from biodiesel product can be assumed as the principal difference in relation to ester group transformation in oil sources to form methyl ester of biodiesel product [15].

Vibration Frequency (cm^{-1})				Integration
BPO	***BPO4***	***BWC***	***BWC4***	
718.71	717.92	718.20	717.13	$-CH_2$ rocking
1454.81	1454.47	1454.60	1454.33	Metyl ester ($CO-O-CH_3$)
1160.1	1159.43	1115.03	1150.24	ester (C-O)
2922.35	2922.15	2922.28	2922.27	-CH2 stretching

Table 2:- The position of main band of FTIR of oil sources and biodiesel product.

On the basis of table 2, it should be noted that the main absorption bands of oil sources (palm and waste cooking oil) and biodiesel product are similar. The FTIR spectra of biodiesel product is undergone a little shift compared to that of oil sources. The similarity in FTIR spectra of biodiesel product and oil sources is due to functional group of triglyceride and methyl ester that have similar structure. The little difference between FTIR spectra of oil sources (palm oil and waste cooking oil) and biodiesel product is caused by difference in chemical bonding existed in oil sources and biodiesel product [16].

B. Flow Rate

The examination of flow rate is used to inform the viscosity of biodiesel product, since the viscosity is reversed to flow rate, in other word, the higher flow rate is an indication of lower viscosity. The flow rates of oil sources are 0.8124 mL/sec. for palm oil and 0.7048 mL/sec. for waste cooking oil. The results show that viscosity of waste

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cooking oil is higher than that of palm oil. The values of flow rate for oil sources and biodiesel are presented in Fig.2 and Fig. 3.

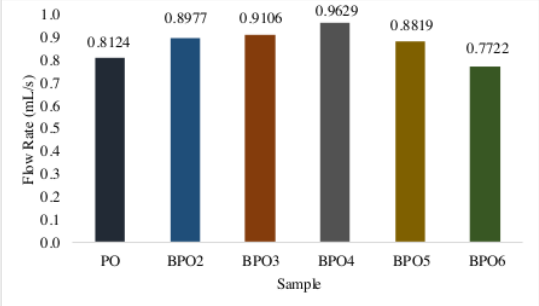

Fig 2:- The flow rates of palm oil (bimoli) and biodiesel products.

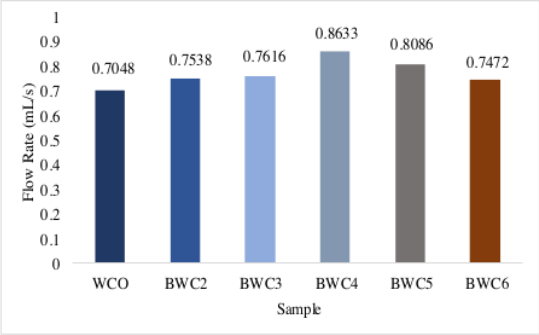

Fig 3:-The flow rates of waste cooking oil and biodiesel products.

Based on the results presented in Fig.2 and Fig.3, in general, the flow rate increased with increasing reaction time until 4h. The flow rate reduced after the reaction time longer than 4h. The increased flow rate until optimum reaction time is caused by triglyceride conversion through transesterification or FFA (Free Fatty Acid) through esterification to yield methyl ester resulting reduced molecular weight and reduced viscosity [17]. The glyceride in palm oil caused high viscosity due to strong hydrogen bonding resulting high density in triglyceride. The shorter reaction time yields lower conversion of glyceride to yield biodiesel resulting higher flow rate or lower viscosity. The waste cooking oil with high FFA, the conversion of FFA to FAME (Fatty Acid Methyl Ester) is occurred through OH substitution from carboxylate with OCH_3 from methanol. The removal of OH group yields increasing flow rate because the hydrogen bonding decreased in the vegetable oil. On the basis of flow rate data, it could be noted that "4h" is the optimum reaction time for biodiesel production from palm oil (bimoli) and waste cooking oil [18].

C. Density

The biodiesel products have also been examined for their density, which are presented in Fig.4 and Fig.5.

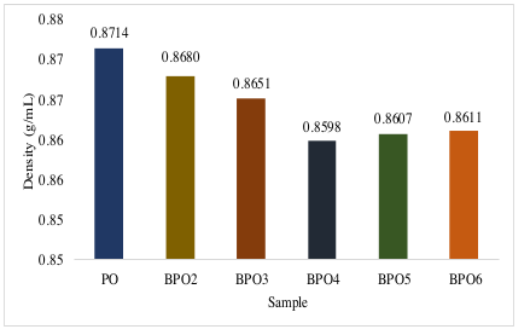

Fig 4:-Density of palm oil and biodiesel products.

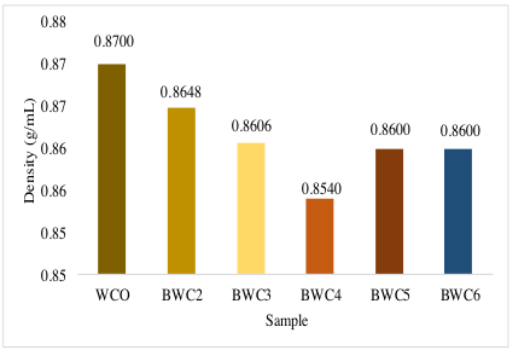

Fig 5:- Density of waste cooking oil and biodiesel products.

The density is also decreased with increasing reaction time until 4h. When the reaction time is longer than 4h the density is increased. The results are consistent with that obtained from flow rate or viscosity. The high density is also caused by water content and methanol in biodiesel due to imperfect separation [18]. Based on the density analysis, the optimum reaction rate for biodiesel production from palm (bimoli) and waste cooking oil using silica-titania catalyst is taken as 4 hours.

D. Acid number and percentage of conversion.

The examination of acid number is used to determine the number of free fatty acid in biodiesel. The results of acid number examination in biodiesel production from palm (bimoli) and waste cooking oil are illustrated in Fig. 6 and Fig. 7.

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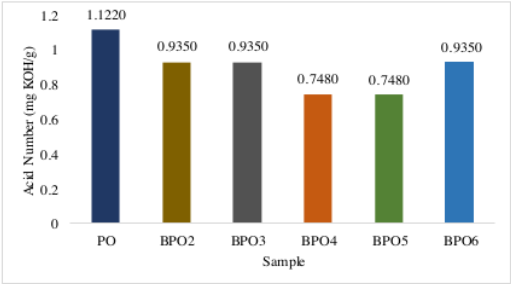

Fig 6:- Acid number of palm oil (bimoli) and biodiesel products.

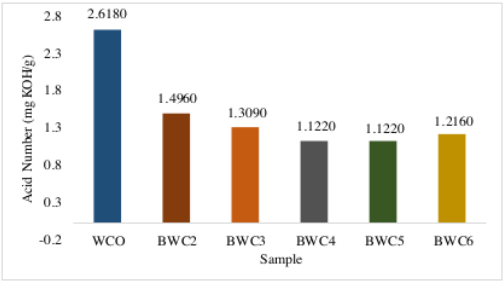

Fig 7:- Acid number of waste cooking oil and biodiesel products.

Fig. 6 and Fig. 7 present the results of acid number examination for biodiesel products and oil sources (palm oil and waste cooking oil). The illustrations show that the acid number of biodiesel products are lower than that of both palm and waste cooking oil. The acid number of biodiesel products decreased with increasing reaction time until 5h and then the acid number of biodiesel products increased again after 5h reaction time. This phenomenon is due to reaction equilibrium achieved and if the reaction time taken longer causing reversed reaction. The longer reaction time reduces biodiesel product and this is caused by hydrolysis of biodiesel in base medium resulting decreased fatty acid methyl ester and the possibility of soap formation. Therefore, immediate separation of biodiesel products from the mixture should be taken and attention on reaction time [2,19]. Table 3 lists the results of acid number, % FFA, and % conversion.

Sample	Acid Number (mg KOH/g)	% FFA (%)	% Conversion (%)
PO	1.122	0.561	
BPO2	0.935	0.468	16.67
BPO3	0.935	0.468	16.67
BPO4	0.748	0.374	33.33
BPO5	0.748	0.374	33.33
BPO6	0.935	0.468	16.67
WCO	2.618	1.31	
BWC2	1.496	0.748	42.86
BWC3	1.309	0.655	50
BWC4	1.122	0.561	57.14
BWC5	1.122	0.561	57.14
BWC6	1.216	0.608	53.55

Table 3:- Percentage of FFA and conversion of biodiesel products.

Based on data of Table 3 conversion of palm oil to biodiesel shows the highest value, i.e. 33.33% at reaction time of 4h and 5h, while conversion of waste cooking oil to biodiesel shows the highest value, i.e. 57.14% at reaction time of 4h and 5h. The biodiesel from waste cooking oil shows higher percentage of conversion compared to that of biodiesel from palm oil. This is due to free fatty acid content in waste cooking oil is higher and can be converted much more to fatty acid methyl ester. In addition, the existence of titanium tetrahedral fraction in silica-titania increasing acidity of catalyst surface in order to convert free fatty acid from triglyceride [13].

The results of physical properties examination of biodiesel products show that reaction time in biodiesel production affected flow rate, density, and acid number of biodiesel products. The optimum reaction time shown by this investigation, i.e. 4h is for both biodiesel products from palm oil and waste cooking oil with different percentage of conversion. Further deduction shows that silica-titania catalyst is an effective catalyst for vegetable oil with FFA content higher than 0.5% [13].

IV. CONCLUSION

The optimum reaction time for biodiesel production from both palm oil and waste cooking oil using silica-titania catalyst is found to be 4h at 65°C. The percentage of conversion for waste cooking oil (57.14 %) is found to be higher than that for palm oil (33.33 %) and this shows that silica-titania catalyst is more effective for vegetable oil with higher FFA content (> 0.5%) in biodiesel production.

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